VACUUM

A Review of Developments in Vacuum Research and Engineering

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VACUUM AS AN 'INERT ATMOSPHERE'

WHY DO WE AIM at pumping down to lower and lower pressures? Essentially because, the smaller the pressure of the residual atmosphere, the less is the danger from interference by its constituents in processes requiring inert surroundings to work satisfactorily. The perfect vacuum of course would represent a complete safeguard against all interference.

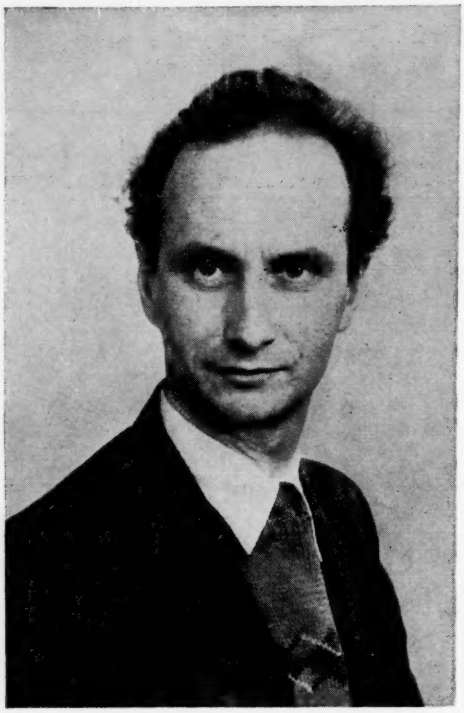
There are projects, mainly in the sphere of research, where it is indeed the aim to eliminate residual matter as completely as possible. For instance, the study of surface phenomena may be seriously impeded by the formation of a deposit of no more than monomolecular thickness. In cases like this, Alpert's ultra-high vacuum techniques have proved their worth. However, there are many instances where the functional requirements of apparatus are satisfied at higher pressures, say 10^{-6} mm. Hg, and extremely low pressures need not be used if interference from the residual atmosphere could be effectively prohibited by other means.

There is no generally applicable interpretation for 'inert atmosphere' in this context. The agencies of contamination, causing 'interference', depend on the nature of the process or the function of the component. One man's meat might be another man's poison. Contamination can originate from the walls of the system, the sealing means such as gaskets, materials present for reasons of the particular process in hand and vapours originating in the pumps used. Organic matter is recognised as a contaminant of major importance with respect to a great number of applications and may be produced from any of the sources mentioned. But in this case the actual quantities present may be appreciably influenced by the choice of pump fluids and gasket materials. As distinct from mercury vapours, organic vapours exposed to heat or the action of an electrical discharge tend to 'crack' and decomposition products may deposit on the surfaces of the system. Also some phlegmatic vapours show a pronounced tendency to adsorb onto surfaces. Thus, if the processed component depends on the proper functioning of an 'active' surface for instance, organic contamination may seriously impair its operational quality. The means of dealing with the problem include baking procedures and the provision of cold traps operated with an expendable charge of liquid nitrogen. But in view of recent trends, these measures are no longer adequate throughout.

In the course of recent years we have witnessed a steady growth of the size of scientific equipment required and consistently rising demands regarding the sensitivity of the components to be manufactured. This trend is fully recognised by the vacuum industry but the countermeasures developed so far are, broadly speaking, merely developments on the lines of long established techniques, rather than by a fundamentally new approach. Thus we have seen the 'comeback' staged by the mercury diffusion pump, preferred today by many operators for the production of high-reliability electronic tubes, and a marked rise of interest in metal gaskets which facilitate the baking of demountable systems. Also, the development of mechanical refrigerators is reported which cool baffles in mercury diffusion pump lines continuously at -100°C, and may eventually displace the conventional liquid nitrogen trap in many cases.

Very recently an original answer, though not fundamentally new, has been forthcoming in a pump based on semi-continuous or continuous gettering action alone, or in conjunction with ion pumping. However, certain limitations in this pumping principle do not permit its use as a remedy for all difficulties arising from 'inert atmosphere' requirements.

An acceptable degree of 'inert atmosphere', amply sufficient to meet all needs in a wide range of industrial and scientific applications, can be obtained without difficulty using the oil diffusion pump. But there is no doubt that the effects of back diffusion of oil vapours, a matter which will be discussed in detail in our next issue, are of great importance, and it is noted with satisfaction that some manufacturers have now started to quote the back-streaming rates of their pumps.



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Dr. Venema was born in Amsterdam in 1916. After completion of his general education at a public school he attended Amsterdam University reading Physics as his main subject. Towards the end of his studies he specialised in cosmic radiation. Under the guidance of Professor Clay he carried out research on the origin of soft electrons in cosmic radiation and eventually wrote his thesis on transition effects, as a result of which he was awarded his degree of Doctor of Physics.

Dr. Venema began his professional career as a teacher at a secondary school but after three years decided to work in industry and joined the Philips Research Laboratories, Holland, in 1946.

His first task as a member of Philips Research Laboratories was an investigation into the performance of electrical contacts, in particular phenomena associated with the transport of contact material in the absence of an arc, known as 'bridge erosion'. This was followed by a study of the mechanism of gold lubrication which was found to improve the operation and service life of electrical contacts.

Only after some years did Dr. Venema concentrate on work connected with vacuum technique. This work arose from problems encountered in vacuum processing at the various factories of the Company and, as it proceeded, gave Dr. Venema ample opportunity of familiarising himself with all aspects of vacuum technology. But some of the problems led to investigations in the field of pure vacuum physics and the article, presented on the opposite page, forms part of such work.

In 1952, Dr. Venema was appointed to take charge of the Research Group dealing with Thermionic Emission. In this capacity he is largely responsible for the development of the latest designs of Philips dispenser cathodes and their successful introduction in industry generally.

the measurement of the pressure in
THE DETERMINATION OF PUMP SPEED

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Summary

THERE IS NO commonly agreed method of determining the speed of a pump. One noteworthy method was proposed by Dayton in 1948 but it has not been accepted generally. Contributions to the problem made by other workers are briefly reviewed followed by a close study of the basic elements of the definition of pressure. Special attention is given to the case where the number-density of the molecules varies along the mean free path. Such variation may occur at low pressures and exists, in particular, in the region of the pump mouth. The actual method of measuring pressure needs more consideration and it is shown that a gauge connected by a tube to the system records the number of molecules per unit area arriving at the gauge-end of the tube. The conditions at the pump mouth have been investigated and an interpretation is given of the results of some measurements with regard to the distribution of the incident molecules. In conclusion, a method of measuring the speed of a pump is proposed which differs from Dayton's method. The new method is based on well-known concepts in the physics of gas flow at very low pressures. It has been put forward before, but simply as a postulate.

Sommaire

IL N'EXISTE PAS de méthode communément acceptée, pour la détermination de la vitesse de pompage d'une pompe. Une méthode, proposée en 1948 par Dayton, et qui n'a pas été acceptée par tous, est à noter. Des publications sur ce problème, faites par d'autres chercheurs, sont brièvement passées en revue, suivies d'une étude scrupuleuse des facteurs de base qui déterminent la définition de pression. On attire spécialement l'attention sur le cas où la densité en nombre des molécules varie, dans la région du libre parcours moyen. Une telle variation peut apparaître aux basses pressions et se produit particulièrement près de l'embouchure de la pompe. La méthode actuelle de mesure de la pression demande plus de considérations et on a montré qu'un manomètre branché au système, au moyen d'un tube, enregistre le nombre de molécules par unité de surface, atteignant la fin du tube du côté du manomètre. On a étudié les conditions relatives à l'embouchure de la pompe et on donne une interprétation de quelques resultats de mesure de pression en fonction de la distribution des molécules incidentes. En conclusion, on propose une méthode de mesure de la vitesse d'une pompe, différente de celle proposée par Dayton. La méthode proposée dans cet article est basée sur des conceptions connues dans la physique de l'écoulement des gas à très basses pressions. Cette conception a été proposée bien avant, mais simplement comme postulatum.

INTRODUCTION

THE BEHAVIOUR OF a vacuum pump is determined to a high degree by the quantity called pump speed. This quantity figures prominently in many publications dealing with diffusion pumps or with pumping procedures in general. At a first glance it is surprising to find that no agreement exists on the ways by which the pump speed is measured. Many cases are known where different authors attach a different meaning to the term pump speed. As a result it is often difficult and sometimes impossible to compare their findings.

The pump speed is closely related to the amount of gas pumped through a vacuum pump in unit time. This amount can be expressed in different units. For the purposes of this investigation the product of pressure and volume will be used to express a gas

* MS. received September, 1956.

quantity. This implies that all measurements of that quantity are effected at the same temperature.

The introduction of the term ' pump speed ' is based on the experimentally determined fact that, in a fairly wide range of pressures, the amount of gas pumped by a diffusion pump is proportional to the pressure p, measured in the region of the pump mouth, and to the time t. It follows that, on dividing this amount by p and t, a quantity is obtained which is constant in that particular pressure range. This quantity is called the pump speed and can be measured in units expressed as the quotient of volume and time. Consistency in the determination of the pump speed depends on consistency in the measurement of the pressure p. It is the aim of this study to clarify the conditions met with, when attempting to measure the pressure in the region of the pump mouth.

The problem of determining the pressure in that region has been the concern of several research workers, notably that of Dayton[1] who made an important contribution to its solution in practice. However in the opinion of the author, his proposals, though widely accepted, do not form the best solution.

We will now discuss Dayton's approach and that of other workers who studied this problem, in greater detail.

Dayton, lecturing on the subject of the speed measurement of diffusion pumps at the Symposium on High Vacuum held at Cambridge Mass. in 1948, drew attention to the fact that the variations in the results obtained by different investigators could be explained by the use of different methods in the experiments. In this connection he stressed in particular the importance of the pressure measurement and stipulated two conditions essential to the achievement of accurate results as follows : (*a*) It is necessary to use a large test dome connected to the pump and, (*b*) the directional effects occurring in pressure measurement must be taken into account.

A test dome with a diameter and a height at least equal to that of the pump diameter simulates more closely practical conditions than, say, a plate placed directly over the pump mouth. In the case of the latter the gas, the speed of which is to be determined, passes through a hole in the plate. This, as Dayton pointed out, facilitates ' beaming ' of the molecules directly into the vapour jet and eliminates random diffusion of the molecules to the extent occurring normally at the mouth of the pump. In this condition the results of pressure measurement carried out directly below the plate will depend largely on the place where the measurement is actually effected. On the other hand, in the case of the test dome, on introducing the gas at a point high up on one side of the dome, proper diffusion will take place and the molecules will be scattered at random.

It is the custom to carry out pressure measurements by means of a gauge which is connected *via* a tube to the region where the measurement is to be taken. The open end of this tube may point in various directions and the results obtained will differ accordingly as has been proved by Dayton giving some figures on these directional effects. These variations will be considerable, in particular, when the place where the pressure has to be measured is chosen to be near the pump mouth. In the case of a good pump the number of molecules leaving the dome and entering the pump will differ substantially from the number of molecules returning to the dome. Therefore, if the open end of the gauge tube points in the direction opposite to that of the molecules entering the pump, the gauge indication will be much higher than in the case, its position is reversed. Dayton came to the conclusion that measuring the pressure in the region of the pump mouth can best be effected by arranging for the gauge tube to be in a perpendicular direction to that of the gas stream. In this way an intermediate pressure is recorded. This is the same procedure as that employed in the ordinary measurement of a moving fluid when the static pressure is to be determined.

The author, while fully agreeing with Dayton on the benefits derived from the use of a test dome, believes, as mentioned already, that the actual arrangement for the measurement of the pressure proposed by Dayton is not the best one. The arguments substantiating this view will be discussed in this article.

Another point also mentioned by Dayton is the fact that some authors introduce the ultimate pressure p_o in the calculation of the pump speed. In this case the amount of the gas pumped through the pump is divid-

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ed by the pressure $(p-p_o)$ and not by the pressure p.

However, as Sears[2] pointed out, this ultimate pressure is in general not determined by one single gas. As the gases behave completely independently of each other in that pressure region, one ought to speak of the ultimate pressure for a particular gas only. In practice, neglecting possible leaks, the ultimate pressure constitutes the pressure of the gases which diffuse back into the pump mouth from the interior of the pump. These gases originate from the pump fluid itself and from its decomposition products. Alternatively they may have been liberated after initial adsorption by the pump walls or they may diffuse through the pump entering on the backing side. It is evident that the ultimate pressure in the accepted sense of the term constitutes the sum of the ultimate pressures of all gases present. Therefore it is wrong to introduce ultimate pressure in a calculation for the pump speed of a special gas. Moreover, there is the point that the different gases behave differently in the gauge itself; thus, the measurement of this pressure presents an additional problem.

The author prefers to base the determination of pump speed on the pressure p directly, not on $(p-p_o)$, choosing a value for p high enough to make p_o, which is in practice of the order of 10^{-6} mm.Hg or less, insignificantly small.

Incidentally, Berry[3] demonstrated the fact that the pump speed of a gas can have a normal value below the level of the ' ultimate pressure '. Berry investigated the pump speed for helium at pressure levels below the ultimate pressure which of course was determined by gases other than helium, and proved that it was the same as it was at pressures higher than the ultimate pressure.

Among other workers who studied the same problem are Witty and Grindley[4] who confirmed, in a short note published soon after the Cambridge Symposium, the observations made by Dayton and expressed the hope that attention would be given to this problem by a larger circle of research workers.

Oatley[5] reports on experiments in 1954 employing a somewhat different method of speed measurement.

Oatley, instead of measuring the quantity of gas pumped through the pump in unit time, kept the total quantity pumped constant and reduced the pump speed by a known amount. The same method was used by Blears and Hill in 1948[6]. The pump is connected *via* an aperture to a large vessel by a short length of large-diameter piping. Means for measuring the pressure in the vessel are available. The aperture between the pipe and the vessel is circular in shape and of variable size. Six different sizes can be provided. The conductance of the various apertures is calculated by Clausing's method. If, in a system of this kind, the amount of gas passing through the pump is kept constant and the conductance of the aperture is varied, then the pressure p in the large vessel is proportional to $(1/S_2+1/S_1)$ where S_2 is the aperture conductance and S_1 the pump speed. Calibration of the manometer in absolute pressure units is not necessary, linearity of pressure indication is sufficient. From the straight line which shows the pressure as a function of $1/S_2$ in the graph, the value of $1/S_1$ can be determined.

While it is not necessary to ascertain the pressures in absolute pressure units in such measurements, it is desirable to know the pressure prevailing at the pump mouth because pump speed is not always independent of pressure. The value of this pressure can be obtained from the graph just mentioned. In this connection it should be stated that it is quite feasible, because the pump speed is defined as a volume per unit time, to measure the speed by comparison with conductance as is done in the Oatley method. However, there are a few points which need clarification. Firstly, how do the values obtained by Oatley's method compare with the values obtained by the Dayton method in the same conditions? Secondly, is the addition $(1/S_1+1/S_2)$ permissible in every case ?

In the ideal case the pipe connecting the vessel, in which the pressure is measured, and the pump should be sufficiently large to allow for the assumption that the distribution of the molecules is homogeneous except for the regions close to the pump inlet and the aperture. In this case there is no pressure drop in the pipe. The molecules which enter the pump mouth obey the cosine law as in Dayton's test dome. Further, if the vessel connected to the pipe is large enough, it can be assumed that the distribution of the

molecules in the vessel, too, is homogeneous. For this set of conditions it is permissible to express the total speed S of the gas leaving the vessel by

$$\frac{1}{S} = \frac{1}{S_1} + \frac{1}{S_2}$$

However, if the dimensions of the pipe and the vessel are not large compared with those of the pump mouth and the apertures, the chance that a molecule will pass into the pump mouth is also influenced by the presence of the tube and the vessel. In these conditions a pressure drop exists in the two components of the apparatus mentioned and the addition $(1/S_1 + 1/S_2)$ is no longer permissible. For this reason Oatley himself insists on employing a short length of a wide-diameter tube. It is clear that the pressure determined by Oatley's method, known as the pressure above the pump, is different from that as defined by Dayton. Only if the pipe used in Dayton's method to connect the pump mouth with the gauge points in the direction away from the pump, *i.e.* into the dome, then the pressure reading will be comparable with the results obtained from Oatley's graph. Consequently, Oatley's method will result in the determination of a lower pump speed than Dayton's method, although the pump and the conditions are the same. This demonstrates the need for a critical investigation into the state of the gas in the neighbourhood of the pump mouth. Such an investigation may assist in obtaining agreement on the procedure to adopt for the determination of pump speed. As a first step in such an investigation the basic elements of the definition of pressure and its measurements will be discussed.

DEFINITION OF PRESSURE AND EFFECT OF NUMBER-DENSITY OF MOLECULES

Before introducing the term pressure we should reflect on the basic elements of its definition. If a gas flows through a pipe, a number of molecules will hit the wall at a given point P per unit area and per unit time. It is well known that this number is determined by the number-density of the molecules in the gas and by their mean velocity with respect to the wall. The molecules hitting the wall at that point exert a continuous force on the wall which, measured per unit area, is called the pressure. Evidently the pressure is a vector.

The point P can be chosen to lie anywhere within the space filled by the gas. Analogous to the previous conception it is now possible to speak of the number of molecules passing through a plane, in which the point P lies, per unit area and per unit time. In order to speak of a pressure distribution in the gas at that point it is necessary to assume that the plane through the point P moves with the mean velocity of the gas at that point. Generally speaking, the mean velocity constitutes a movement of the gas in the neighbourhood of the point P, which is additional to the thermal motion of the molecules. Here, as in the previous case, the pressure exerted on this plane is a vector and, generally speaking, will depend on the direction of the plane. In particular, we will consider the normal component of the pressure on the plane. In the case, we have three planes passing through the point P, perpendicular to each other and all moving with the mean velocity of the gas, then the mean hydrostatic pressure at P is defined as the mean of the normal components of the pressure on these planes. Given certain conditions, the quantity which is introduced in this manner is, as can be shown by calculation, the mean of the squares of the thermal speeds. Its magnitude is, of course, independent of any specific arrangement of these planes. The mean hydrostatic pressure is an essential feature of the kinetic theory of gases. This theory states that the mean of the squares of the thermal speeds is proportional to the temperature. It is thus possible to obtain immediately the equation of state for an ideal gas.

In the following discussion $\mathbf{C}$ is the thermal velocity of the molecules. Their velocity relative to the plane is $\mathbf{C}'$ with the normal component C_n'. The actual velocity of the molecules is $\mathbf{c}$. $\mathbf{C}=\mathbf{c}-\mathbf{c}_0$, where $\mathbf{c}_0$ is the mean velocity of the gas at point P. The velocity distribution function will be denoted by f.

The number of molecules passing the surface element $\mathrm{d}S$ in the time $\mathrm{d}t$ in one direction is given by[7] :

$$\nu \, \mathrm{d}S \, \mathrm{d}t = \mathrm{d}S \, \mathrm{d}t \int_{C_n'>0} C_n' f \, \mathrm{d}\mathbf{C} \qquad (1)$$

ν is the number of molecules passing unit area in unit time. The validity of this expression is based on the assumption that dt is so small that the molecules passing the surface element in that time suffer no collision with other molecules.

The number-density n, which is the number of molecules per unit volume, is given by

$$n = \int f \, \mathrm{d}\mathbf{C} \qquad (2)$$

If n is constant in the region of the space where the molecules passing through the surface element are coming from, then

$$\nu = n \, \overline{C_n'} \quad (C_n' > o) \qquad (3)$$

If, however, n varies appreciably in this region, *i.e.* if n varies noticeably along the mean free path of the molecules, this variation must be taken into account while assessing the number of molecules which pass dS. This applies to the conditions encountered in the neighbourhood of the pump mouth. At normal pressures, changes of n over a distance equal to the mean free path are negligible.

For the pressure $\mathbf{p}_n$, exerted in one direction on the surface element dS of the gas, the following relation applies :

$$\mathbf{p}_n \, \mathrm{d}S \, \mathrm{d}t = \mathrm{d}S \, \mathrm{d}t \int C_n' \, m \, \mathbf{c} \, f \, \mathrm{d}\mathbf{C} \qquad (4)$$

where m = the mass of the molecules.

The same expression is valid for the pressure on the wall. Here again, the formula can be simplified, if n is constant in the region mentioned, and Equ. (4) becomes :

$$\mathbf{p}_n = n \, m \, \overline{C_n' \mathbf{c}} = n \, m \, \overline{C_n} \, \mathbf{C} \qquad (5)$$

The sum of the normal components of the pressures exerted on any three orthogonal planes is

$$n \, m \, \overline{C^2}$$

and the mean of the normal pressures on these planes is thus

$$\tfrac{1}{3} \, n \, m \, \overline{C^2}$$

This magnitude is called the mean hydrostatic pressure in the gas at that point.

If n is not constant, the number-density in the three directions will be different and the simple expression $n \, m \, \overline{C^2}$ for the sum of the normal pressures no longer applies. However, in this case the quantity

$$p^* = \tfrac{1}{3} \, n \, m \, \overline{C^2} \qquad (6)$$

is well defined at the point P. Instead of the mean hydrostatic pressure this quantity can be used to define the gas situation at P together with the density ρ or the temperature T. Generally speaking, the forces acting on the planes through the point P and the quantity p^* are not related in this case.

It will be clear that both the number of incident molecules and the pressure which they exert on the surface element are influenced in the same way by variations of n. For, when a molecule has reached dS, the pressure exerted is determined solely by its mass m and its velocity **c.**

$C_n' > \mathrm{O}$ in Equ. (3) means that only half of the molecules present in one cubic centimetre contribute to ν. In order to express the mean normal component $\overline{C_n'}$ in C' a simple integration is needed, the result of which is $\overline{C_n'} = \frac{1}{2} C'$. Thus it is evident that the number of molecules passing through the surface element is

$$\nu = \tfrac{1}{4} \, n \, \overline{C'} \qquad (7)$$

which can also be stated in the form of the well-known expression

$$\nu = \tfrac{1}{4} \, n \, \overline{C} \qquad (7a)$$

when the gas is at rest with respect to the surface element.

For the case where n cannot be assumed to be constant, a general expression for ν cannot be given. Such an expression would, of course, depend on the variations of n in the region adjacent to dS. The same holds for the pressures exerted on the wall or any plane in the gas.

In order to get some idea of the effects of a variation of n at distances up to that equalling the mean free

path, on the number ν, the following hypothetical case will be considered. Assuming n depends only on one co-ordinate, *e.g.* the x co-ordinate

$$n(x) = n(o) + x \frac{\partial n(o)}{\partial x} + \ldots \qquad (8)$$

where $n(o)$ represents the number-density at the point P, the surface element dS lying in the yz plane. The mean free path will be expressed by L. The probability that a molecule, found at a distance r from P and travelling in the direction of dS, reaches dS without having suffered a collision is given by exp. $(-r/L)$. As it is assumed that all molecules move with the mean velocity $\overline{C'}$ relative to dS, the number of molecules that will pass dS in one direction is

$$N = \int_0^{\infty} \int_0^{\pi/2} \frac{n(x) \exp(-r/L) \sin I \cos I \, dI \, dr}{2} dS \qquad (9)$$

After inserting $x = r \cos I$ and Equ. (8) in Equ. (9) we integrate and obtain

$$N = \left[\tfrac{1}{4} n(o) L + \tfrac{1}{6} \frac{\partial n(o)}{\partial x} L^2 + \ldots . \right] dS \qquad (10)$$

This result shows, among other things, that the mean distance, over which the molecules have travelled before reaching dS, is L, which is to be expected. To travel this distance the molecules require the time $dt = L/\overline{C'}$. It follows from Equ. (10) :

$$\nu = \tfrac{1}{4} n(o) \overline{C'} + \tfrac{1}{6} \frac{\partial n(o)}{\partial x} L \overline{C'} + \ldots \qquad (11)$$

The first term on the right-hand side of this equation is the well-known expression (see Equ. (7)). The second, however, shows the influence of the variation of n. The ratio of the first two terms is

$$\frac{2}{3} \frac{\partial n(o)}{\partial x} \frac{L}{n(o)}$$

which can be expressed as

$$\frac{2}{3} \frac{\partial p}{\partial x} \frac{\alpha}{p^2} = - \frac{2}{3} \alpha \frac{\partial \frac{1}{p}}{\partial x}$$

where $\alpha = \dfrac{L}{p} = 5 \times 10^{-3}$ cm/mm. Hg for air.

If now

$$\frac{\partial p}{\partial x} = 10^{-4} \text{ mm. Hg/cm.}$$

and $p = 10^{-3}$ mm. Hg.

this ratio becomes 1/3; this shows the great influence of the variation of n on the number of incident molecules. It is also evident that the influence becomes smaller as the pressure becomes higher. The condition expressed in Equ. (8) is not likely to occur in practice. Nevertheless, the calculation given above shows clearly the great importance of variations of n and gives an indication of what can be expected under practical conditions.

This discussion will have made it clear that the conditions prevailing in the pump mouth can only superficially be compared with those existing in a pipe through which a liquid or a gas at higher pressures is flowing. In the latter case there is the static pressure which is the pressure within the moving fluid and the dynamic pressure which is due to the motion of the mass of the fluid as a whole. The static pressure can be measured by means of the gauge tube with its open end pointing in a direction perpendicular to the flow. The value of the total pressure is obtained if the gauge tube is placed in a position pointing in the direction opposite to that of the flow and the dynamic pressure is obtained by subtracting the value of the static pressure from that of the total pressure. In the region of the pump mouth the molecules have only their thermal motion and a flow of gas can only be caused by a difference in number-density. It would be better, therefore, to compare these conditions with those in a pipe, through which gas flows at normal pressures. Here, too, total pressure consists of two components, a static and a dynamic pressure, but as the pressure in the gas is determined solely by the number-density and the thermal velocities, differences in the static pressure will be strictly related to differences in number-densities. However, in the high pressure case the change in number-densities in the pipe may be small and all molecules will have the

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same extra velocity in the direction of the pipe axis. This shows that there is a fundamental difference between the two sets of conditions and a comparison does not assist in clarifying the picture with regard to the region at the pump mouth.

MEASUREMENT OF THE PRESSURE

As the pressure is generally defined as a force acting on 1 cm^2 its measurement involves, properly speaking, the measurement of a force. However, only in very few cases is a force actually measured. Of all the pressure gauges normally used in the low pressure range only the diaphragm gauge can be considered an instrument capable of measuring a force.

Furthermore it is evident from the discussion in the previous section that in general an instrument in a fixed position will not give an indication of the pressure within the gas. At low pressures, however, where the movement of the molecules is purely thermal, the measurement of the gas pressure can be effected by means of such an instrument.

Besides the choice of the type of gauge, there is the question of the connection of the gauge to the system. Only a few gauges exist which can measure the pressure precisely at the point where it has to be determined. Usually a connection has to be provided by means of a tube between the region which contains the test point and the gauge. If the gauge has no pumping action of its own, the gas in this tube is at rest. If, however, the gauge has a pumping action, it is a well-known fact that great care has to be exercised in evaluating the readings because a pressure difference exists between the two ends of the connecting tube. But, even if the gauge has no pumping action and the gas in the tube is at rest, there may still be a pressure difference between the ends of the connecting tube because of a difference in temperature. At low pressures in particular, this effect may be appreciable. At high pressures, where the mean free path is small compared with the dimensions of the tube, the pressure is the same at all points. The case of the intermediate pressure range is more difficult. There is no general theoretical answer to this question but experimental results are available which might help to determine the pressure difference[9].

The indication given by the gauge at one end of the connecting tube must be considered a measure of the number of molecules, passing through an area of 1 $cm.^2$ of a plane perpendicular to the tube axis at the other end of the tube. This indication does not necessarily constitute a measure of the pressure in the gas at this point; it merely gives the number of incident molecules. It was shown in the previous section that the number of molecules incident on a surface element dS and the pressure which they exert on this element, both depend to the same degree on the number-density in the neighbourhood of dS. Therefore, the measurement of the number of incident molecules can also be considered a measure of the pressure which these molecules would exert on a surface element, placed perpendicular to the tube axis at the end of the tube. Temperature effects must of course be taken into account.

In the following discussion we will assume that no temperature difference exists between the ends of the tube which connects the gauge to the system. Below, a few of the gauges normally employed for the measurement of pressures less than 0.01 mm. Hg are given, and comments are added on the actual quantity recorded by the instrument in each case.

The *McLeod gauge* measures a certain number of molecules brought into a known volume, the capillary, and determines the pressure which these molecules exert in this volume. Thus, the gauge records in fact the number of molecules per cm^3 present in the connecting tube. At the system-end of the connecting tube this number of molecules per cm^3 constitutes the number of molecules migrating into the system which is the same as that which enters the tube at the other end.

The *diaphragm gauge* measures directly a pressure. If it is installed at the point where the pressure is to be ascertained, it gives a direct reading but, if it is connected to the system by means of a tube, the pressure in the tube is measured and this is determined by the number-density of the molecules.

The *heat-conductivity gauge* measures the number of molecules which strike a surface heated to a known temperature. Thus, if the gauge is mounted at the

point where the pressure is to be ascertained it gives an indication of the number of molecules passing through the region where the heated surface, generally a wire, is situated. The number of molecules which will strike the heated surface depends upon the method of mounting. In theory, it must be possible to obtain direct information on the number of molecules which enter a volume element from all directions. If the gauge is connected to the system by means of a tube, the number of molecules striking the heated surface is proportional to the number-density in the tube.

The *viscosity gauge* is similar to the heat-conductivity gauge in that respect. Here, too, the number of molecules striking a surface is measured.

The *Knudsen gauge*, like the McLeod gauge, is connected to the system by a tube. It records the number-density.

The *ionisation gauge* can be installed directly at the point where the pressure is to be determined. It measures then the number of molecules passing a certain region determined by the geometry of the gauge and the voltages applied. Molecules from different directions can enter this region and here, too, the indication of the gauge can be a measure of the number of molecules entering a volume element from all directions. Again, if the gauge is connected by means of a tube, it indicates the number-density.

CONDITIONS AT THE PUMP MOUTH

Having concluded the discussion on the concepts, number of incident molecules and pressure measurement, we will now turn our attention to the conditions met with, when determining pressure in the vicinity of the pump mouth.

The number of molecules incident on a plane situated in that area depends upon the direction of that plane. In the same way the pressure exerted on the plane depends on that direction. But it has not yet been finally decided which direction is to be used when measuring the pressure in that area for the purpose of calculating pump speed. Is it feasible to determine the quantity p^* (see Equ. (6)) and use it in this calculation ?

According to the conclusions drawn in the theoretical discussion in the second section of this paper and assuming that there is a sizeable difference in the number-density along a distance equal to the mean free path, knowledge of the number of molecules incident on a surface element in a plane through the point P does not constitute information on the number-density existing precisely at the point P. We only have some information on the mean number-density applicable to the vicinity of that point extending along a distance equalling the mean free path. These are the conditions we have to deal with in the region of the pump mouth. Here, for instance, we may find a density gradient of the order of 10^{12} mol./cm.^{4} at a density of, say, 15×10^{12} mol./cm.^{3} which means a mean free path of about 10 cm. in air. Thus, over a distance equalling one mean free path we have a variation of number-density of approximately 30%. Therefore, if, in our measurements, we use an instrument connected to the pump mouth by means of a tube measuring the number of incident molecules, it will not be possible to conclude from the reading what the actual number-density is at the pump-mouth end of the connecting tube. However, this value must be known in order to facilitate the determination of the quantity p^*.

It might be supposed that measuring the number-density in different directions may yield a method for the calculation of the mean value which could be identified with the actual number-density at the mouth. However, in view of the arbitrary distribution in the vicinity of the pump mouth, such a calculation could not possibly give a true answer. For this reason Dayton's proposal cannot be used. Also, the molecules coming from within the pump and entering the connecting tube will, generally speaking, not have the same velocity distribution as the molecules moving in the opposite direction. The temperature of the regions within the pump will be higher and thus the total velocity distribution of the molecules will be unknown. Therefore, the determination of $\overline{C^2}$ will present additional difficulties.

At this point we should consider some experiments which have been carried out to obtain more information on the distribution of the number of incident molecules in the vicinity of the pump mouth. In all

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these experiments air was the gas used.

In the first series of experiments a 4–inch. diffusion pump with a speed given as 200 l./sec. by the supplier was employed. A test dome, almost entirely spherical in shape, was connected directly to the pump. A small spherical probe with openings in various directions was placed in the pump mouth. By closing all but one of these openings it was possible to measure the number of incident molecules in any particular direction. The particular opening employed in any one experiment was always situated in the plane of the pump mouth. Fig. 1 gives the details of this arrangement. The results of this first series of experiments are summarised in Table I. Within experimental

Table 1. Effects of Direction on Gauge Readings

Direction of Measurement	*Reading of the McLeod Gauge* $\times 10^{-4}$ *mm. Hg*	
	Throughput: 9.2 mm. Hg cm³/sec.	*Throughput: 102 mm. Hg cm³/sec.*
Tube-end points into the test dome ↑	0.61	5.53
→	0.52	4.80
Tube-end points into the pump ↓	0.44	4.25

error the indication obtained in the direction normal to the pump axis (→) is the mean of those in the other two directions (↑ ↓). These results show clearly that, while objections may be raised against the principle of Dayton's proposal, the error incurred in the determination of the number-density, while working to Dayton's method *i.e.* with the open end of the test tube pointing perpendicularly to the pump axis, has been quite small in this particular case.

The problem of the velocity distribution still exists but, because the contribution of the faster molecules will be small in this case, their influence may be negligible. The nozzle of the pump used in these experiments was 7 cm. beneath the mouth of the pump. Therefore, the disturbance caused by the pump was not very great. If the pump nozzle had been closer to the pump mouth, it is probable that the difference between the mean of the indications in the two opposite directions and the indication perpendicular to these directions would have been greater.

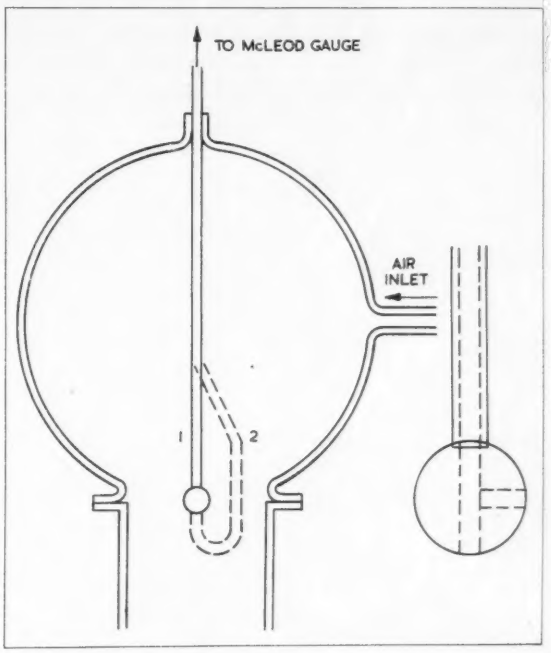

Fig. 1. Experimental arrangement for the measurement of the directional effects on the pressure indication in the pump mouth. Pos. 1 for the directions ↓ and →, Pos. 2 for the direction ↑.

A second series of experiments was arranged in order to determine, how far inside the test dome directional effects could still be traced. The results of these experiments showed a noticeable difference between the mean of the two indications in the two opposite directions and the indications in the direction normal to the former two.

The second series of experiments were carried out with a 2¼–inch. pump, the speed of which was given as 70–80 l./sec. As before, measurements were taken in three different directions but, in addition, the measurements were taken at different distances from

the pump mouth inside the test dome. The test dome was spherical as before and had a diameter of 15 cm. but no spherical probe was used. The use of the more complicated spherical probe was discontinued as it had been found in the initial experiments that the employment of the normal connecting tube gave the same pressure readings. The air inlet into the dome was half-way between the top of the dome and the pump mouth. Fig. 2 shows the results of the McLeod readings.

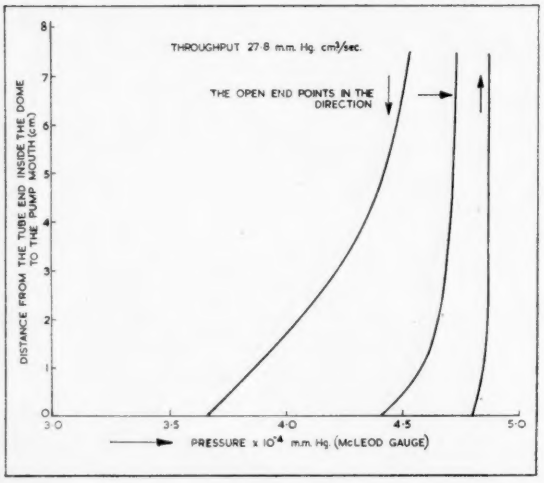

Fig. 2. The influence of the position of the tube-end on pressure indication.

In the second series of experiments the distance of the point from the pump mouth, at which the measurements were taken, was found to have a marked effect on the readings particularly in the ↓ direction. On the other hand, in the ↑ direction the influence of the distance was hardly detectable. At the pump mouth the mean of the readings in the opposite direction was 4.22×10^{-4} mm.Hg, whereas the readings in the perpendicular direction were 4.40×10^{-4} mm. Hg. The difference between these two values is outside experimental error. At points within the dome the difference is smaller, as was to be expected.

Another problem which is connected with the subject of this series of experiments, is the optimum size of the test dome which will ensure that the effects of the opening of the pump mouth can be considered negligible. In order to find an answer to this problem, domes of various sizes were connected to the $2\frac{1}{4}$–inch. pump. Domes of a size very much larger than the pump mouth were spherical; the smaller ones, however, had to be of semi-spherical shape. In these experiments the connecting tube of the gauge was joined to the dome half-way between the top and the pump mouth but did not project into the dome. With this arrangement, the number of molecules incident on the wall of the dome was determined. The air inlet was joined to the dome at the same height as the gauge tube but 90° apart. The results of these experiments are given in Table II.

Table II. The Influence of the Diameter of the Test Dome on Pressure Readings

Readings of the McLeod Gauge : × 10^{-4} mm. Hg
Diameter of the Pump Mouth = 5.7 cm.

Throughput mm. Hg cm³/sec.	*Diameter of the Test Dome (cm.)*				*Mean Free Path cm.*
	6	10	15	24	
7.95	1.25	1.34	1.32	1.33	40
27.8	4.38	4.80	4.80	4.74	10
85.6	14.7	14.8	14.7	14.5	3.4

It appears that only in the case that the mean free path is small compared with the diameter of the pump and the dome, the pressure readings for all domes are the same. When the mean free path is equal to, or larger than, the diameters of these apparatus, the readings taken in experiments using the smallest dome differ by about 6% from those taken in experiments using the larger domes. It can be concluded that a reasonably accurate measurement of the number of molecules incident on the wall of the dome is obtained if the diameter of the dome employed in the measurements is about 3 × the diameter of the pump mouth.

Where the mean free path is very long, it is also possible to calculate the effect of the size of the pump opening on the pressure reading obtained at the wall of the dome. If this condition is not fulfilled, it is easier to establish this effect by means of experiment.

PROPOSED METHOD OF DETERMINING PUMP SPEED

So far no attempt has been made to give details of a feasible method of pressure measurement facilitating the determination of pump speed. From what has been said before it is evident that the determination of the quantity p^* at the pump mouth, defined by Equ. (6), presents great experimental difficulties.

The conditions prevailing at the pump mouth resemble closely the conditions existing at one end of a pipe with a steep pressure gradient where the pressure is kept very low. The problem of gas flow at very low pressures has been investigated by several authors[7, 10, 11] and there is basic agreement on the method by which the amount of gas flowing through a pipe should be calculated. In these conditions the molecules travel independently of each other. It is assumed that the pipe connects two infinitely large vessels the pressures of which are p and zero. If the pressure is larger than zero at the particular end of the pipe, the total flow can be considered to consist of two separate flows of this kind moving in opposite directions. The number of molecules which pass through the pipe end from the pressure side in unit time can be easily ascertained. It is given by the product of the cross section of the pipe and v. The next step in the calculation, *i.e.* the determination of the number of molecules flowing through the pipe amounts, in fact, to a determination of the probability of a molecule which enters the pipe at one end, passing through the pipe and escaping at the other end. The problem of density changes in the neighbourhood of the pipe-opening can be neglected in the calculation. The only physical quantity introduced is the number of molecules incident on the pipe-opening. As we are concerned with a rarified gas, this number is solely determined by the number-density of the molecules in the infinitely large volume. But the number-density is proportional to the pressure in that volume, and this can be easily determined as long as the measurement is not taken too closely to the pipe-opening. The quantity of gas flowing through the pipe per second is then given by

$$Q = Fp \qquad (12)$$

where F is the conductance of the tube, determined only by its dimensions.

The conditions just described are almost completely identical with those prevailing at the pump mouth, as long as the pump is connected to a sufficiently large volume. Here, too, molecules enter the pump and escape into the interior of the pump. There is a difference however, as some of the molecules return as a result of collisions with the pump wall and components. Moreover, these molecules will have a different velocity distribution, because of the raised temperature of the pump components in question. But, it has been demonstrated experimentally that Equ. (12) applies also to the condition at the mouth of a pump.

The test dome provides the large volume required. As stated above, its diameter should be three times that of the pump mouth. The pressure p in the dome is determined by measuring the number of molecules incident on the wall by connecting the gauge to a hole in the wall of the test dome. As long as temperature differences are absent, the gauge will give a direct reading of the pressure in the dome. Alternatively, the gauge can be placed inside the dome, at a distance sufficiently far away from the pump mouth[6]. It is then possible to express the quantity of gas removed from the dome by the pump in unit time by :

$$Q = Sp \qquad (13)$$

where S is the pump speed.

At very low pressures this method will give the same results as obtained by measuring the pressure in the pump mouth with the open end of the gauge tube facing the test dome. The small difference in the neighbourhood of the mouth shown in Fig. 2 is probably due to the fact that the mean free path was not long enough. It was only about 10 cm. in the particular experiment.

The method of determining the pressure described above varies appreciably from that proposed by Dayton and is essentially the same as that employed by Oatley. In order to be successful the pipe and vessel used by the latter author must be of sufficiently large dimensions. The fact that Oatley's original method provides for a comparison of the pump speed with

conductances of known magnitudes is a minor detail.

CONCLUSIONS

The above investigation permits the conclusions :

(*a*) The values obtained in measuring the pressure at the pump mouth depend on the direction, in which the measurements are taken.

(*b*) The gas condition prevailing at the pump mouth can be defined with the aid of the quantity :

$$p^* = \tfrac{1}{3}\, n\, m\, \overline{C^2}$$

(*c*) The assessment of n and C^2 is very difficult and may well be impossible.

(*d*) A comparative study of gas flow through a pipe reveals that a test dome of large dimensions to be connected to the pump is required for this type of measurement.

(*e*) The pressure measured in the test dome should be used for the determination of the pump speed.

The method described gives consistent values for the pump speed under a variety of conditions. This can best be demonstrated by way of example. Let us suppose that the speed of a large pump is to be de ermined. As a first step the pump is connected to a test dome of comparatively large dimensions. The pump mouth is then partly closed with a very thin plate leaving a circular hole for the passage of the gas. As this hole is reduced in size, the throughput is decreased so that the pressure in the test dome will undergo no change. But the pressure in the tract between the mouth and the nozzle of the pump will steadily decrease. When the hole has become very small, the pressure on the pump side of the plate will be negligible. Even in this case it is still possible to speak of a speed of the modified pump : It is given by $\pi r^2 \tfrac{1}{4} \overline{C}$, an expression which defines the conductance of a circular hole with the radius r. In Dayton's method the value obtained for the speed will be exactly twice as much as before because the pressure measured in the hole perpendicular to the pump axis is only half of that measured in the dome. Such contradictory results are of course absent in the method recommended in this paper.

The method described has one general disadvantage, *i.e.* the values of the pump speed obtained will invariably be smaller than those determined by the Dayton method. Thus, at first sight, it will not appear very attractive to the manufacturer who is used to determining pump speeds by the Dayton method.

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References

1 DAYTON, B. B., *Industr. Engng. Chem.*, **40,** (1948), 795-803. ' Measurement and Comparison of Pumping Speeds '.

2 SEARS, G. W., *Rev. Sci. Instrum.*, **20,** (1949), 458. ' Ultimate Vacua of Diffusion Pumps '.

3 BERRY, C. E., *Rev. Sci. Instrum.*, **20,** (1949), 835. ' Pumping Speed of Diffusion Pumps below Limiting Pressure '.

4 WITTY, R. and GRINDLEY, J., *J. Sci. Instrum.*, **25,** (1948), 351-352. ' Measurement of Pumping Speeds '.

5 OATLEY, C. W., *Brit. J. Appl. Phys.*, **5,** (1954), 358-362. ' The Experimental Determination of the Speed of a Vacuum Pump and of Components of a Vacuum System '.

6 BLEARS, J. and HILL, R. W., *Rev. Sci. Instrum.*, **19,** (1948), 847-851. ' The Speeds of Diffusion Pumps for Gases of Low Molecular Weight '.

7 CLAUSING, P., *Ann. Phys., Lpz.*, **12,** (1932), 961-989. ' The Flow of Gas at Very Low Pressure through Tubes of Various Lengths '.

8 CHAPMAN, S. and COWLING, T. G., *The Mathematical Theory of Non-Uniform Gases.* (Cambridge University Press, London, 1952).

9 RUSCH, M. and BUNGE, O., *Z. Tech. Phys.*, **13,** (1932), 77-81. ' Sources of Error in Pressure Measurements with Liquid Air Traps '.

10 KNUDSEN, M., *Ann. Phys., Lpz.*, **28,** (1908), 75-130. ' Laws of Molecular Flow and Internal Viscosity Flow of Gases through Tubes '.

11 SMOLUCHOWSKI, M. Von, *Ann. Phys., Lpz.*, **33,** (1910), 1559-1570. 'Zur Kinetischen Theorie der Transpiration und Diffusion Verdünnter Gase '.

PERFORMANCE CHARACTERISTICS OF BARIUM GETTERS

with particular reference to their application in thermionic valves

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Summary

THE PERFORMANCE CHARACTERISTICS of pure barium getter deposits (bright mirrors only) evaporated from a barium-aluminium alloy have been investigated. In the Introduction an outline is given of the task of the getter in all phases of the production and working life of a thermionic valve. The capillary method was used in the experiments but whereas previous workers operated this test with the pressure in the manifold being constant, the author modified the equipment to facilitate accurate operation of the test with the pressure in the getter chamber constant. The results thus obtained are independent of specific design features of the test employed. It is proposed to assess getter performance in terms of 'instantaneous absorption capacity' expressed in litre micron per second. This facilitates in addition the calculation of the total absorption capacity of the getter by integrating the curve giving the instantaneous absorption capacity values obtained by measurement. If presented in this manner the results of such investigations are of immediate assistance to industrial practices. The behaviour of the getters towards a number of pure gases has been re-examined employing the revised capillary method.

Sommaire

LES CARACTERISTIQUES de performance des dépôts de baryum pur, des getters (dépôts brillants seulement) évaporés à partir d'un alliage d'aluminium et de baryum, ont été étudiées. Dans l'introduction, on donne un aperçu du rôle du getter dans toutes les phases de la production et de la vie d'une valve thermionique. La méthode capillaire fut employée pour les expériences, mais tandis que les premiers chercheurs opéraient avec pression constante dans le manifold, l'auteur modifia l'équipment pour faire des opérations plus précises, ayant ainsi une pression constante dans la chambre à getter. Les résultats ainsi obtenus ne dépendent pas des caractéristiques propres à l'appareil employé. On propose d'évaluer l'action d'un getter comme 'capacité instantanée d'absorption' exprimée en micron litre par seconde. Ceci facilite en plus le calcul de la capacité totale d'absorption du getter en intégrant la courbe de la capacité instantanée d'absorption obtenue par mesures. Presentés de cette facon, les résultats de telles recherches sont d'un intéret immédiat dans l'application industrielle. Le comportement des getters en présence de differents gaz purs a été re-étudié, en employant la méthode capillaire revisée.

INTRODUCTION

GETTERS HAVE BEEN EMPLOYED for more than 30 years in the electronics industry, and more specifically in the manufacture of thermionic valves. There are many patents covering getter materials and various forms of their application but, broadly speaking, these and a large proportion of the few investigations published so far are essentially concerned with problems of the type encountered in the practical usage of getters. Proper scientific information on the actual mechanism of gettering is lacking. It is the merit of the Japanese investigators, Ota, Kobayashi and Furuya, and Wagener in England to have initiated comprehensive basic research into getter performance[1-8].

Extending the service life of valves without impair-

*MS. received May, 1956.

ing specified electric characteristics is one of the major aims of the valve industry today[9] and manufacturers have begun to realise that a better understanding of the getter mechanism will greatly assist in this task.

The author, in co-operation with Dr. Origlio, has started methodic investigations on the subject in his own laboratories. Our investigations are based on, and form an extension of, Wagener's experiments and have so far yielded a series of values for the gas quantities absorbed by a getter and certain fresh information on the gettering mechanism which, we believe, is of immediate practical use to the engineer concerned with thermionic valve production.

The data presented in this paper is restricted to the performance of barium getters and should not be considered complete or final. We intend to add to the information given here in future contributions to this journal, when fresh results become available in the course of our research work.

TASK OF A GETTER

The 'life' of a sealed-off radio valve can be divided into three phases:—

(1) Production of the operational pressure in the valve,
(2) Ageing of the valve, and
(3) Operation of the valve under service conditions.

The getter, introduced into the tube during manufacture, has the purpose of absorbing any gas present at any of the three phases.

Modern manufacturing methods provide for the valve to be sealed off before the getter is evaporated. This is the correct procedure as the evaporation of the getter results in an immediate reduction of the pressure in the valve to a level below that prevailing in the pumping system and, if the pumping system were not sealed off at that stage, gas would be drawn into the valve. Usually the valve is sealed off at 1×10^{-3} mm.Hg while the operational pressure required to be attained in the valve by the getter is of the order of 1×10^{-7} mm.Hg. In actual fact, sometimes an even better vacuum, of the order of 1×10^{-9} mm.Hg is obtained[10].

The action of the getter in this, the first phase, is fast, *i.e.* its initial absorption rate down to pressures ranging from 9×10^{-6} mm.Hg to 1×10^{-6} mm.Hg (according to the nature of the gases dealt with) is high, subsequently the rate slows down. The quantity of gas absorbed by the getter in the first phase is comparatively small. For instance, take the case of a valve of 0.05 l. volume, sealed off at 1×10^{-3} mm.Hg; here, operational pressure is obtained after the absorption of no more than 0.05 ml. of gas. This represents only a fraction of the total absorption capacity of a modern barium getter. Therefore, if a valve is rejected at this stage on the grounds of poor vacuum, this must be due to one of two possible causes: (*a*) leakage to atmosphere, or (*b*) faulty (or damaged) getter. There are in fact instances where getters, such as the old-type (non-stabilised) pellet getters, liberate more gas than they can absorb.

Up to a few years ago it was generally believed that the purpose of a getter is confined to this phase. The belief may have been caused by the assumption that the activity of the getter is ineffective in the second and third phase, or by a lack of appreciation of the problem of gas evolution during the service life of the valve. Therefore, as it became apparent that the absorption capacity of the getters in general amply covered the requirements of the first phase, there was no need for basic studies of getter performance in the two subsequent phases. Consequently, little is known on getter behaviour during these stages and more research work is called for on such questions as the origin, nature and amount of gases present during the ageing stage (Phase No. 2). Similarly, more information is required on gas evolution during the service life of the valve, *i.e.*, during Phase No. 3. The latter task may prove particularly difficult. Most likely only statistical methods will yield useful results and this will require a close check on operational conditions in each case.

However, some light was thrown on the conditions prevailing in Phase No. 3 by investigations published recently by various workers and discussed in detail below. Here we mention only Deb's[11] study on the decay of cathode emission. He shows that the poisoning of cathodes is effected by gases liberated con-

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tinuously from the anode as a result of electron bombardment. This could lead to the suggestion of plotting gas evolution from the anode and establishing a similar record for the absorption activities of the getter. A comparison of the two graphs might assist directly in determining whether the getter in question is capable of maintaining the required low pressure in the valve or, failing that, whether it is capable of preventing the pressure from rising to a level dangerous to cathode emission. But this is not yet a practical suggestion because it is not known what constitutes the critical pressure with respect to cathode poisoning. All we know is that the cathode temperature is an important factor in this connection and that a cold cathode is damaged more easily than a hot cathode[12].

Finally, there is the important question: Is the poisoning of the cathode in certain circumstances a reversible phenomenon and, if so, what are the conditions leading to reversible poisoning?

Lack of intimate knowledge of the gettering mechanism has led to many misconceptions. For instance, many workers believe that a barium getter with an intact mirror is always efficient. The fact is that mirror appearance is no indication at all. There are cases where the absorption capacity of the mirror is completely exhausted but its appearance is unchanged. Whatever the appearance of the mirror, a getter can only absorb a certain amount of gas. Many studies have been made of the gas content of the various valve materials and the results of these studies can be used to estimate the absorption capacity of the getter. But there are no rules covering all contingencies. The performance of some valves, operated intermittently, shows that the getter absorbs continuously all the gas evolved during their operation for periods extending over years. Others subjected to heavy duty may show oscillations in their performance after an initial run of a few hours. These phenomena may be only of a transient nature but nevertheless have to be dealt with and are likely to arise from fluctuations of the pressure in the valve which, in turn, originate from unsatisfactory getter performance.

A good example of the benefits which can be derived from a study of operational conditions and gettering mechanism in Phase No. 3 presents itself in the field of modern transmitter valves. These valves are fitted with thoriated tungsten cathodes of an especially delicate type. Long service life of these tubes was at last achieved, employing three zirconium getters, each operative at a different temperature and absorbing a specific gas at each temperature level[13].

THE BARIUM GETTER

Barium is the material most commonly used for gettering radio valves, transmitter valves (up to 500 W), vacuum rectifiers, t.v. and oscilloscope tubes and radar equipment. Barium getters can be obtained commercially from various sources[14]. Below, the more important types of barium getters are listed and a brief indication of their individual characteristics is given in each case:—

(1) *Pure Barium.* The barium is filled into small iron, nickel or copper tubes. The tubes are sealed off and subsequently cut into pellets, 4-5 mm. long. For use the pellets are mounted on a nickel flag and evaporated by means of h.f. heating. This type of getter is now obsolete due to its lack of stability when exposed to air while being mounted in the valve.

(2) *Barium Stabilised by Alloying with Aluminium.* The material, a 50 : 50 barium-aluminium alloy, is sealed off in steel tubes flattened on one side. For use the tubes are mounted on nickel loops and heated by means of h.f. Barium only is evaporated.

(3) *Reactive Barium Compounds.* The material, a mixture of barium carbonate or beryllate with tantalum reducing powder and, in some instances, a binding substance is applied to nickel or molybdenum flags. The flags are mounted on nickel loops and heated by means of h.f. or d.c. Barium only is evaporated. The reaction of these getters is exothermic causing appreciable evolution of gas.

(4) *Barium Stabilised by Alloying with Refractory Metals.* A special-purpose getter to ensure good adhesion to the walls of the vessel, etc.

The material evaporated is pure barium.

(5) *Barium Stabilised by Alloying with Metals Evaporating at Low Temperatures.* Calcium is alloyed to the barium in order to obtain a low evaporation temperature. Magnesium used for the purpose in the past was found to cause the mirror deposit to peel.

(6) *Barium-Alloy Rings.* The properties of these getters are not yet fully explored. The results of investigations, still in progress, will be reported in due course. The material evaporated is pure barium.

In this paper we are concerned exclusively with the behaviour of barium getters belonging to Group (2) which is the type mostly used in receiver valve manufacture at the present time.

COMMENTS ON PREVIOUS WORK

The action of getters was studied first some 20 years ago. The methods employed in the early studies were rudimentary, the lowest pressures obtainable were about 10^{-4} mm.Hg and instruments lacked the required accuracy. It is no surprise, therefore, that the results were often misleading. For instance, it was held to be proved that getters are ineffective if operated in a high vacuum. When it was found that getters can be instrumental in lowering the pressure in valves to 10^{-10} mm.Hg and maintain this pressure for a long period, the qualification was added that getters will remain effective at low pressures if an ionising discharge is present. This interpretation of getter characteristics has still, today, many adherents in the valve industry.

Wagener, investigating the effect of a discharge on the gettering activity of barium deposits, initially obtained results which appeared to confirm the above theory: Deposits close to the discharge showed high absorption activity. However, in a later experiment where the deposits were connected at electrical potentials different from those of the getter itself, contrary to expectations no changes in absorption activity with ionising electron current could be observed. An attempt[3] to explain these observations on the lines of established thought was abandoned, when, in a final series of experiments employing a Knudsen gauge, positive proof was obtained that the ionising action of a discharge has no influence on the absorbing power of a getter. These findings[7], arrived at in 1954, indicated clearly that a new approach to the problem of getter mechanism was required and, incidentally, demonstrated that ionisation gauges can be used in such investigations without distorting the results. The phenomena which, in Wagener's early experiments, appeared to confirm the old theory may have been due to temperature effects as it is known that the temperature of mirror deposits located near a discharge is higher than that of deposits further away.

The sequence of events just described is typical for research developments in that field. Generally speaking it can be said, and will be shown below, that the greater part of the data available is of little use to the practical man because certain factors having a bearing on the performance of getters, have not received sufficient attention in the past.

EXPERIMENTAL SET-UP

The methods and the apparatus used in the present investigations are essentially the same as those employed by Wagener[3–5]. Certain minor modifications resulted in a reduction of the time required for an experimental run to one day at the most. The method is based on the now almost traditional principle of measuring getter activity in terms of the flow of gas through a capillary.

The equipment, shown diagramatically in Fig. 1, consists of a manifold, a chamber housing the getter under test and the capillary connecting the two vessels. A silicone-oil diffusion pump with a speed of 60-70 l./sec. giving an ultimate pressure of 5×10^{-7} mm.Hg is connected to the manifold. The getter chamber varied in size from that equivalent to a miniature thermionic valve to the equivalent of a large t.v. tube. The capillary is fitted to a (mechanical) valve of special design which contains no metal component and permits quick change of capillaries to suit individual test conditions. The pressure is measured with the help of two ionisation gauges connected to the manifold and the getter chamber respectively.

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The pressure in the manifold can be controlled by a needle valve which serves at the same time as a means of admitting the gas, the absorption of which is to be tested. The provision made for the quick change of capillaries has the advantage that the size of the capillary can be selected to suit the pressure range employed and the gas studied under each test. In this manner we can make certain that operations are always carried out in the range of high sensitivity of the gauges and that the pressure difference between the two chambers can be reduced if required to keep within the stable range of the pumping speed of the diffusion pump employed. Capillaries of various diameters were used in the experiments but their length-to-diameter ratio was 50 : 1 in every case. The whole plant is made of Pyrex glass and degassed at 450°C for two hours before an experiment is started.

In operation, gas coming into contact with the surface of the getter is absorbed. As a result the pressure in the getter chamber is reduced to a value below that in the manifold and gas begins to flow from the manifold into the getter chamber. The amount of gas passing per unit time through the capillary E is a measure of the activity of the getter under test and is determined by the formula

$$E = F\,(p_m - p_g) \qquad (1)$$

The conductance of the capillary F is given by

$$F = \frac{66}{\sqrt{M}} \cdot \frac{d^3}{l + 4d/3} \cdot 10^3 \qquad (2)$$

M = Mass of the gas
d = Capillary diameter (cm.)
l = Capillary length (cm.)
p_m = Pressure in the manifold, and
p_g = Pressure in the getter chamber

E is thus determined by calculating the conductance of the capillary and measuring p_m and p_g.

REMARKS ON EXPERIMENTAL PROCEDURE

Many of the factors influencing getter performance vary in practice and it is hoped that proper allowance for these conditions can be made at some future stage of our investigation. However, in the work reported here certain of these factors have been kept at a fixed value and are common to all experiments, in order to obtain truly comparative results of general significance.

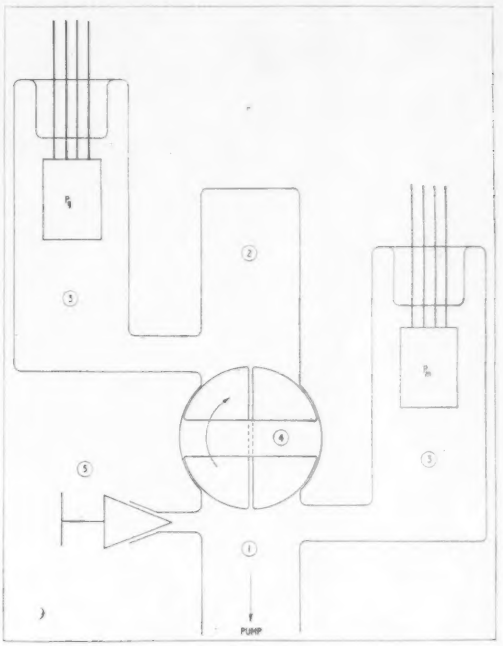


Fig. 1. Diagram of the experimental equipment. *Legend:* (1) Manifold, (2) getter chamber, (3) ionisation gauges, (4) valve carrying capillary, (5) needle valve. The valve (item 4) facilitates fast evacuation of the equipment.

The getter deposits investigated are pure barium, evaporated from a barium-aluminium alloy, see Group (2) on page 286.

The ratio of the weight of the evaporated barium to the area of the deposit under test is throughout about 0.2 mg/cm². This ratio corresponds to a theoretical thickness of solid barium of 0.6 micron. But, as can be seen in Table I, variations in the thickness of the deposits have no effect on the absorption of the gas except in a few cases.

All measurements relate to a temperature of the mirror deposit of 20°C unless otherwise stated.

As the gas liberated by the getter during evapora-

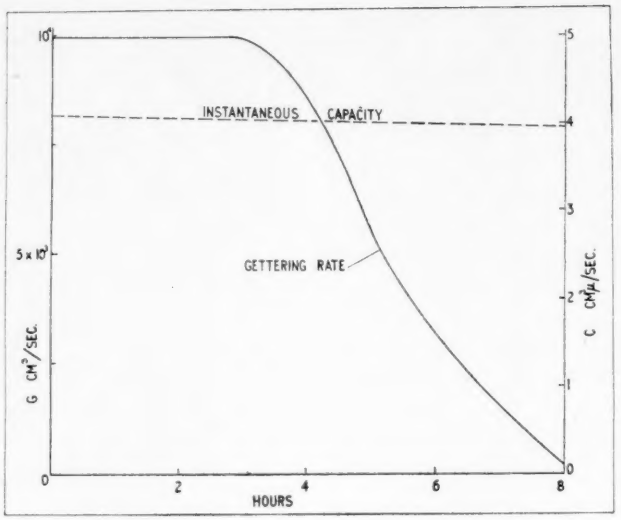


Fig. 2. Absorption capacity and gettering rate of carbon dioxide determined, keeping the pressure in the manifold constant at 4.4×10^{-4} mm.Hg. Area of the barium mirror 150 cm.^{2}

Fig. 3. Instantaneous capacity of carbon monoxide determined, keeping p_m constant at 5.7 $\times 10^{-4}$ mm.Hg, using two different capillaries: (1) Having a conductance of 34 cm.^{3}/sec. and (2) having a conductance of 11 cm.^{3}/sec.

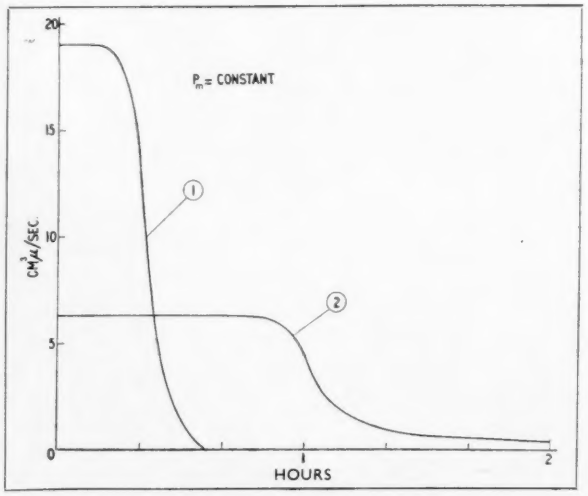
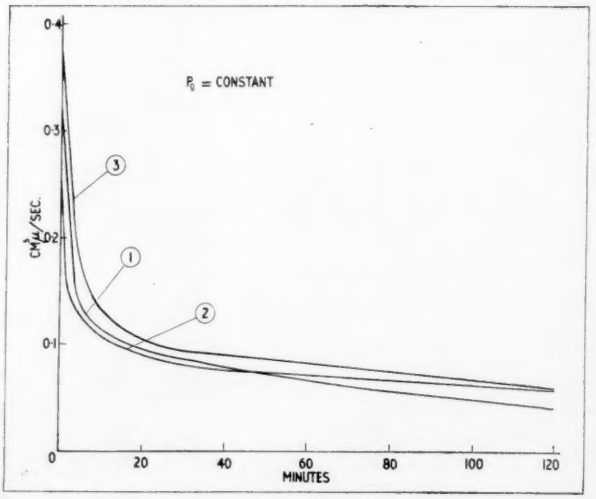


Fig. 4. Instantaneous capacity of dry air determined, keeping the pressure in the getter chamber constant at 3×10^{-4} mm.Hg, using three different capillaries: (1) Having a conductance of 2.75 cm.^{3}/sec., (2) having a conductance of 5.5 cm.^{3}/sec. and (3) having a conductance of 14.4 cm.^{3}/sec.



tion may affect the accuracy of the observations in the subsequent experiments, the precaution was taken of evaporating the getter while the chamber was still being pumped. The pressure at that stage was 5×10^{-5} mm.Hg. Thus, all data given below refers to bright mirrors. The problem of the dark mirror, which incidentally is always bright on the inside, has been ignored for the time being because the writer is convinced that valve manufacture is already sufficiently complicated without warranting any additional special efforts in order to obtain efficient dark mirrors. This does not mean, of course, that a study of the properties of the dark mirror should be neglected altogether.

The evaporation of small getters took 6-8 seconds and that of large getters 10-12 seconds.

All getters investigated were thoroughly degassed prior to use. As mentioned already the vacuum system was baked before an experiment started and the pressure in the getter chamber was invariably of the order of 3 to 6×10^{-7} mm.Hg at this stage.

The gases employed in the experiments were of commercial purity and were admitted to the system after drying by phosphorus pentoxide. In the case of water vapour care was taken to eliminate dissolved air.

SIGNIFICANCE OF GETTERING RATE AND GETTERING CAPACITY

As stated before, the performance of getters is assessed by this method in terms of their pumping power. Following Wagener this pumping power may be determined by measuring the reduction of the pressure in the getter chamber after evaporation of the getter, keeping the pressure in the manifold at a known constant value. A large pressure difference indicates a high pumping power and a small pressure difference indicates a low pumping power of the getter.

Wagener had satisfied himself at an early stage of his work that the volume of gas absorbed by a getter in a radio valve is small compared with the total absorption power of the getter and he decided[2], therefore, that the essential task is to promote speedy absorption of the gases originating in the various parts of the valve by attracting them towards the getters, *i.e.*, he considered the speed of absorption, termed the gettering rate G, the essential element of the pumping power.

G is expressed by[5]

$$G\,p_g = F\,(p_m - p_g) \tag{3}$$

$$G = F\,\frac{p_m - p_g}{p_g} \tag{4}$$

when

$$p_m \gg p_g$$

$$G = F\,\frac{p_m}{p_g} \tag{5}$$

This theory is further developed in a later publication[4] where an expression is given for the final residual pressure obtained in a valve through the action of the getter as follows:—

$$V\,\frac{dp}{dt} = E(t) - p_g\,G(t) \tag{6}$$

where

$E\,(t)$ is the amount of gas instantaneously evolved from the various parts of the valve during operation,
$G\,(t)$ is the instantaneous speed of absorption by the getter, and
V is the volume of the valve.

This is integrated, and by choosing suitable limits the residual pressure is expressed as:

$$p = \frac{E\,(t)}{G\,(t)} \tag{7}$$

Equ. 7 constitutes the final argument in this approach and signifies that as long as $E\,(t)$ is constant, the value of the ultimate residual pressure obtained in a tube by getter action is solely governed by the gettering rate.

Thus, practically all data available on gettering rates has been determined by the capillary method, keeping the pressure in the manifold constant.

The initial experiments carried out in the laboratories of the writer were conducted on the same lines. Employing a large barium getter and measuring the

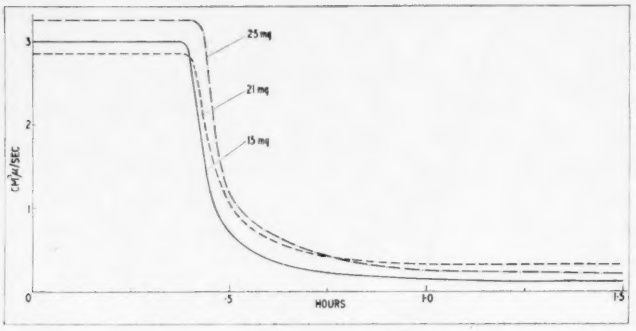


Fig. 5. Values of the instantaneous capacity for carbon dioxide obtained at constant $p_g = 5 \times 10^{-6}$ mm.Hg, with three barium deposits of equal area = 10 cm.² but different weights.

gettering rate for carbon dioxide we obtained the values shown by the solid line in Fig. 2. It was then decided to check on the actual quantities of gas absorbed by the getter during the same period and this produced the values indicated by the dotted line in Fig. 2. It will be noted that the dotted line indicates nearly unimpaired absorption power of the getter at a time when the gettering rate suggests complete exhaustion. In a fresh series of experiments employing capillaries of various diameters the character of the capacity curve changed completely. The maxima obtained rose steadily as the diameter of the capillaries was increased.

These results demonstrate that the values of the gettering rate, obtained in experiments where the pressure in the manifold is kept constant, really depend on the pressure above the getter. A change of the latter from, say, 10^{-4} mm.Hg to 10^{-5} mm.Hg will increase the gettering rate by a factor of 10 but the amount of gas passing through the capillary in unit time will remain practically the same. In experiments conducted with p_m = constant the results obtained are applicable only in conditions identical to those of the specific test equipment and procedure employed.

The observations suggest the use of the alternative method of manipulating the capillary test, *i.e.* keeping the pressure in the getter chamber constant during the experiment. This method and certain results obtained by it are mentioned by Wagener in the article[5] published in this journal but as far as we know the method has not been made use of on an appreciable scale in subsequent work.

The following considerations will show that our aim of tracing and presenting data on getter action in a form generally applicable and of immediate practical interest can indeed be achieved by employing the method of p_g = constant.

Analogous to Equ. 1 the maximum instantaneous quantity of gas $Q_{inst.}$ passing through the capillary is:—

$$Q_{inst.} = F\,(p_m - p_g) \qquad (8)$$

As long as the method employed is that of p_m = constant, the amount of gas supplied to the getter chamber will rise only up to a certain figure and will remain constant at that level, dependent on the pressure maintained in the manifold and on the conductance of the capillary employed in the particular test. Also, the effect of p_g on the quantity Q_{inst} passing into the getter chamber is appreciable only as long as its value is nearly the same as that in the manifold. On the other hand, if the process of absorption is controlled by maintaining a constant pressure above the getter, the instantaneous quantity of gas entering the getter chamber is independent of the conductance properties of the capillaries and of the previous limitations on the flow. Now, the only governing factor is the instantaneous absorption capacity of the getter itself.

Keeping p_g constant adds to the difficulties met with in the experimental procedure. It requires continuous and undivided attention on the part of the operator as he must vary the value of p_m, instantly, in step with any fluctuations of p_g. However, if the right choice of capillaries is made, no undue difficulties are experienced.

The conditions just discussed can be conveniently demonstrated by reference to Figs. 3 and 4 giving values obtained with p_m = constant and p_g = constant respectively. As can be seen the dimensions of the capillary have no material effect on the results in

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Fig. 4. By arranging for a series of experimental runs, each carried out at a different constant p_g, truly comparative values are obtained for the gas under test as long as the same temperature is employed in all experiments.

In view of what has been said before we concentrated at an early stage on measuring the gas quantity instantaneously absorbed by the getter. This quantity is identical with the instantaneous amount of gas passed through the capillary and, in physical dimensions, corresponds to a given rate of gas evolution in a valve. We called this quantity 'instantaneous absorption capacity' and defined it—as the evolution rate—in cm^3/sec. adjusted to the pressure of 1 micron Hg. The curves we obtained for this quantity give a much clearer indication of the action of the getter than the curves obtained for the gettering rate and, in addition, facilitated the evaluation of the total absorption capacity of the getters, independently of the pressures prevailing in the vessel during measurement, by integrating the curves.

The total absorption capacity is normally given in litre micron, *i.e.* it represents the total quantity of gas, a getter can absorb in litres of gas at a pressure of 1 micron Hg. Appreciable confusion is created by the procedure, still followed by many workers today, of relating throughout the absorption properties of barium mirrors to the weight of the evaporated barium instead of the area of the deposit. There are many gases, for which the absorption by the deposit is merely a surface phenomenon and in all these cases gettering capacity is proportional to the surface area of the deposit. By way of example, reference should be made to Fig. 5 giving absorption values for carbon dioxide. Three tests are recorded, carried out on deposits of equal area but of different thickness. The graph clearly demonstrates that the thickness of the mirror has no effect on the plotted values and that the total capacity determined by the integration of the curves is equally unaffected. On the other hand Fig. 6 shows the absorption curve of two mirrors, one of 5 $cm.^2$ and the other of 10 cm^2. The amount of barium evaporated to produce the two deposits was the same. The capacity values determined for the two mirrors are related to each other by the same ratio as the surface areas of the deposits. Similar observations have been made in experiments with hydrogen, nitrogen and air. Therefore, in the case of all these gases we shall speak in future of total capacity per sq. cm.

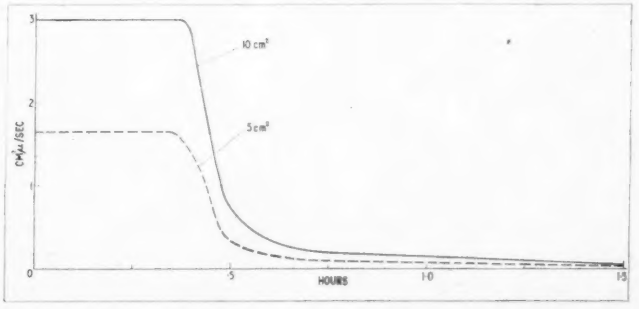

Fig. 6. Values of the instantaneous capacity for carbon dioxide obtained at constant $p_g = 5 \times 10^{-6}$ mm.Hg with two barium deposits of equal weight but different areas.

EXPERIMENTAL RESULTS

General

Before we give detailed results on the behaviour of barium getters towards individual gases, we will discuss below observations, made in the course of the experiments, regarding their absorption characteristics in general.

Total Absorption Capacity. Table I gives the total absorption capacities of barium getters for various gases determined at a temperature of the deposits of 20°C. A close examination of these values will give rise to some doubt as to whether it is correct to assume that the absorption capacity of a getter is ample under all practical conditions. We already mentioned a study[11] which proved that gas is evolved continuously during the operation of a valve. Other investigations point in the same direction. For instance, Jacobs and Wolk[15] have observed that during a period of 90 minutes immediately following the activation of the cathode at 900°C, a quantity of 5.5

litre micron of gas was evolved and, when the measurements were repeated after a further two hours, it was found that as much gas had been liberated again. Considering that the surface of the deposit in a modern miniature valve will not exceed 5 cm²., it is obvious that the absorption capacity of a getter is fully exploited in many cases. Indeed, frequently, the values shown in Table I would not suffice and proper working of the valve would not be ensured but for the fact that the deposit operates at a temperature appreciably higher than that at which we took our measurements. In this connection reference should be made to the results obtained by Hibi and Ishikawa[12] who investigated possible remedies against the decay of cathode emission. They showed that raising the temperature of the cathode would not stop the decay of cathode emission but is of benefit indirectly as it causes the temperature of the barium deposit to rise and thus increases its efficiency.

Table I. Total Absorption Capacity of Bright Barium Mirrors

Gas	*Quantity Absorbed*		*Influence of the Weight of Barium Evaporated on Absorption Capacity*
Oxygen	50	l.μ/mg.	—
Moisture	35	l.μ/mg.	—
Hydrogen	4.48	l.μ/cm².	no
Carbon Dioxide	0.60	l.μ/cm².	no
Carbon Monoxide	0.72	l.μ/cm².	no
Nitrogen	0.33	l.μ/cm².	yes
Dry Air	0.64	l.μ/cm².	yes

Instantaneous Absorption Capacity. The discrepancies between results of the measurements at p_m= constant and those at p_g= constant have been discussed on page 291. In the case of p_g=constant the capillary rate and the instantaneous absorption capacity have the same trend, *i.e.* the values obtained experimentally for the instantaneous absorption capacity C_{inst} are strictly proportional to those obtained for the gettering rate G.

This is to be expected, as analogous to Equ. 4

$$G = F\,\frac{p_m - p_g}{p_g} = \frac{C_{inst}}{p_g} \qquad (9)$$

From a practical point of view absorption capacity curves are to be preferred as they facilitate determination of the total getter capacity by integration.

Peculiarities of Getter Characteristics. In our experiments employing the method p_g = constant we noticed two phenomena of special interest :

(*a*) An abrupt cessation of the absorption activity of the getter if exposed to carbon monoxide and dioxide gases, and

(*b*) An apparent maximum of the gettering rate for each gas applicable to a certain pressure level in the high vacuum range.

(*a*) is a confirmation of facts reported in a previous publication[5]. Figs. 7 a-c and Figs. 8 a-c give the instantaneous absorption, capacities and gettering rates for carbon monoxide carbon dioxide, hydrogen, nitrogen and air at pressures (p_g) of 3×10^{-5} mm. Hg, 9×10^{-6} mm.Hg and 5×10^{-6} mm.Hg respectively. All getter deposits employed in the experiments had the same surface area=1 sq. cm. The graphs show very different absorption characteristics for carbon monoxide and dioxide gases than for all other gases. Whereas hydrogen, nitrogen and air are absorbed at a steadily declining rate, carbon monoxide and dioxide are absorbed at first at a constant rate but after a short period all absorption ceases abruptly. The lower the pressure at which the tests are conducted, the less marked is this phenomenon but it is never completely absent.

(*b*) Figs. 9b, 10b, 11b, 12b and 13b show gettering rates for a number of gases measured at various pressures p_g. In the case of nitrogen for instance, it will be noticed that the initial values of the gettering rate increase, the lower the operational pressure, see the curves for $p_g = 3\times10^{-4}$ mm.Hg, 3×10^{-5} mm.Hg and 9×10^{-6} mm.Hg. But if the operational pressure is as low as 5×10^{-6} mm.Hg this trend appears to be

reversed: The initial values of the gettering rate are reduced and the slope of the curve is appreciably less. Similar conditions have been observed with hydrogen and carbon dioxide. The significant pressure in the case of air is 2.5×10^{-6} mm.Hg and in the case of carbon monoxide 3×10^{-5} mm.Hg.

Details

There follows a detailed discussion of the results we obtained in our experiments exposing barium deposits to a variety of pure gases at low pressures.

Oxygen. The total absorption capacity of barium deposits for oxygen was found to be 50 litre micron per milligramme. But the getter action deteriorates rather suddenly after an initial period. This is due to the formation of a barium oxide layer on the surface of the deposit. A gramme molecule of barium oxide has a volume of 25.4 c.c. as compared with 37.5 c.c. in the case of pure barium, *i.e.* the barium oxide layer has a smaller volume than the original pure barium deposit. As a result the barium oxide layer is highly porous and permits the oxygen to penetrate the barium oxide layer and to reach the pure barium deposit underneath. Similar observations are recorded by Wagener[5]. The formation of a barium oxide layer is indicated by a progressive change in the appearance of the original deposit. In all our tests the metallic appearance of the mirror had disappeared entirely after a while.

Fig. 14 gives a record of the behaviour of two getter deposits of different weight. As can be seen, initially the instantaneous absorption capacity (and therefore the gettering rate) is nearly the same but the total absorption capacity depends on the amount of barium deposited.

Of all the gases present in the valve during operation oxygen is considered the most dangerous because it poisons the cathode beyond remedy. The adverse effects of oxygen are obviously due to its affinity for free barium, the presence of which is considered essential for the maintenance of good emission from the cathode. Oxygen is evolved at a very slow rate but its evolution continues for a period during the service life of the valve. For this reason the getter must have a high total absorption capacity and must remain effective for a long time. Action as best as possible must be taken to prevent poisoning of the getter by the deposition of layers which will stop the oxygen from reaching the barium surface.

Accordingly, it is of the utmost importance that valve components receive proper treatment to minimise oxygen absorption prior to assembly. The hydrogen employed in the reduction process should be pure and the equipment leak-tight, otherwise the impurities may offset the effect of the treatment. Of the workers[11, 15, 16] who have investigated the composition of the atmosphere in valves and its effects on cathode emission, Levergon and Shepherd[17] reported a study of particular interest in this connection. Observing operational conditions in valves generally they found that barium strontium oxide cathodes liberate appreciable amounts of barium oxide which deposit on the anode. Due to the continuous electron bombardment the anode evolves oxygen. Unless getter action is effective this free oxygen returns to the cathode and forms an oxide with free barium on its surface. This effect is assisted by the presence of barium in the evaporated state during operation and is aggravated if there is a poor vacuum in the valve.

Moisture. The average total absorption capacity for water vapour was found to be 35 litre micron per milligramme. In practice widely different values have been obtained in the various experimental runs, possibly for the reason that compounds other than $Ba(OH)_2$ are formed in the presence of moisture. One gramme molecule of $Ba(OH)_2$ has a volume of 38 c.c., almost the same as that of pure barium. It is unlikely therefore that the barium mirror becomes porous. It is not quite clear how the water penetrates into the interior of the deposit. In several tests it was noted that part of the mirror surface never assumed the milky and iridescent appearance, normally associated with the conversion into barium hydrate. This observation seems to agree with our measurement of total absorption capacity since, theoretically, 130 litre micron of moisture must be absorbed to convert 1 milligramme of barium into barium hydrate. The instantaneous absorption capacity for moisture appears to depend on the surface area and not on the thickness of the mirror, see Fig. 15.

Values of the Instantaneous Capacity

Determined for Various Gases, Keeping p_g = const.
(Barium Mirror = 1 cm.²)

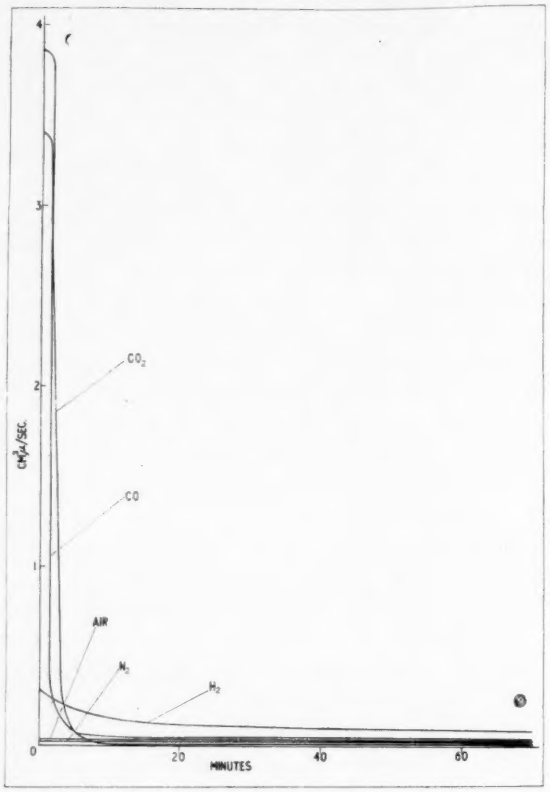

Fig. 7a. $p_g = 3 \times 10^{-6}$ mm.Hg.

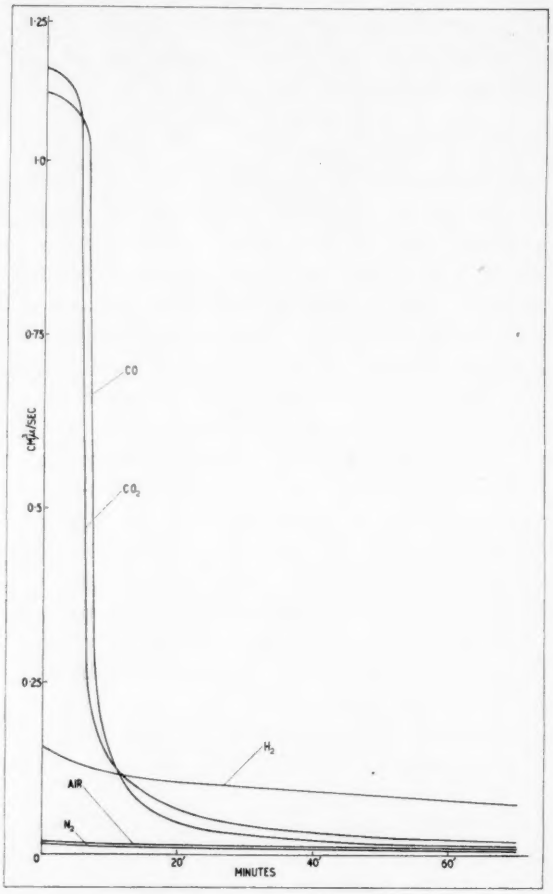

Fig. 7b. $p_g = 9 \times 10^{-6}$ mm.Hg.

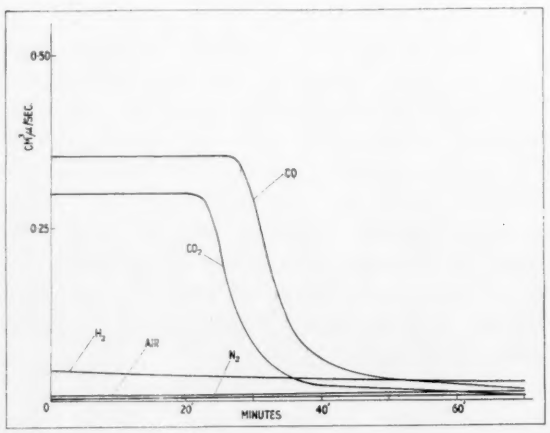

Fig. 7c. $p_g = 5 \times 10^{-6}$ mm.Hg.

It appears to be the general consensus of opinion that moisture is rarely present in sufficient quantities to cause poisoning of the cathode. This is essentially attributed to the improvement in the performance of modern pumps[15]. Any moisture present in the valve after the sealing-off stage may have originated from an excessive amount of carbon dioxide which will initiate the reaction $CO_2 + H_2 = H_2O + CO$ as hydrogen is usually available in abundant quantities. Another potential source of water vapour is mica[16]. During the heat treatment of the valve at about 600-700°C, mica may release water vapour and if the quantities of moisture involved are large, the mica

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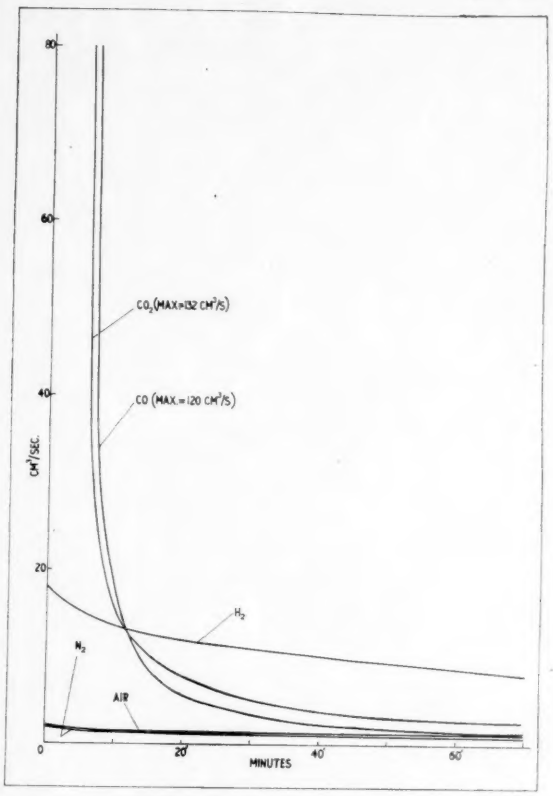
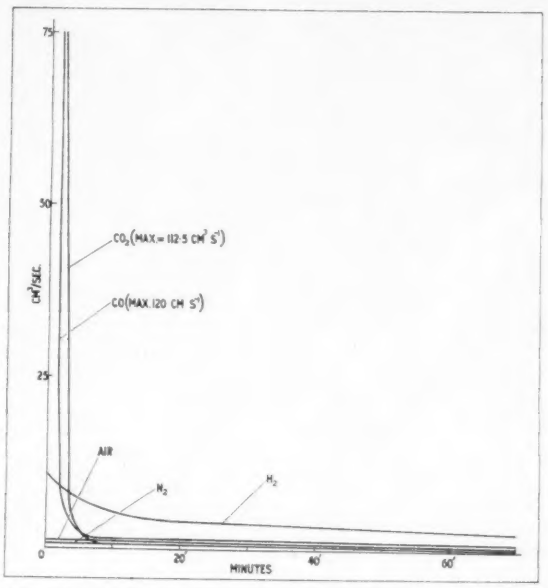
Values of the Gettering Rate

Determined for Various Gases, Keeping p_g = const.
(Barium Mirror = 1 cm.²)





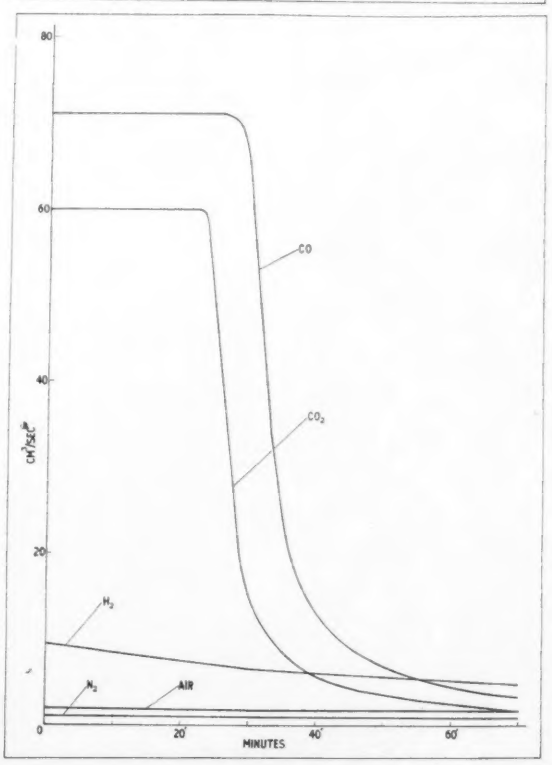


Fig. 8a. (Top right) $p_g = 3 \times 10^{-5}$ mm.Hg.

Fig. 8b. (Above) $p_g = 9 \times 10^{-6}$ mm.Hg.

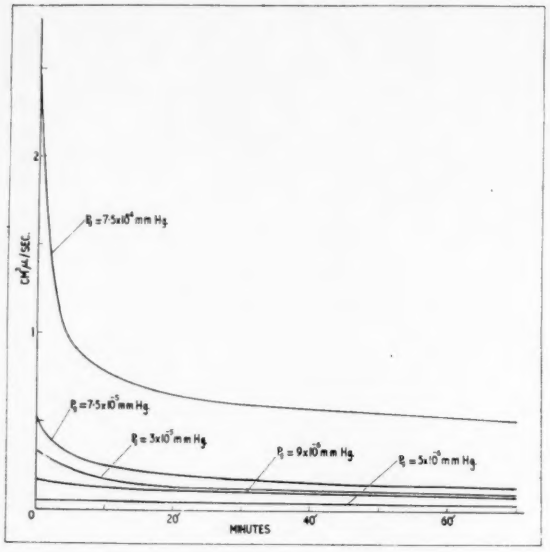
Fig. 8c. (Right) $p_g = 5 \times 10^{-6}$ mm.Hg.

will swell and this will lead to the rejection of the valve in the production test.

Hydrogen. Our measurements show a total absorption capacity for this gas varying from 30 to 45 litre micron per milligramme. Theoretically the figure should be 135 litre micron per milligramme. Thus, we cannot confirm that the gas attacks the whole of the barium mirror deposit as was stated previously by Wagener. Also, the compound formed in the process is Ba (H_2), one gramme molecule of which has a volume of only 33 c.c. Therefore, pores should develop in the mirror while absorption is proceeding and this, in turn, should promote the absorption of large amounts of

Values of the Instantaneous Capacity

Obtained at Various Pressures with a Barium Mirror of 1 cm.²

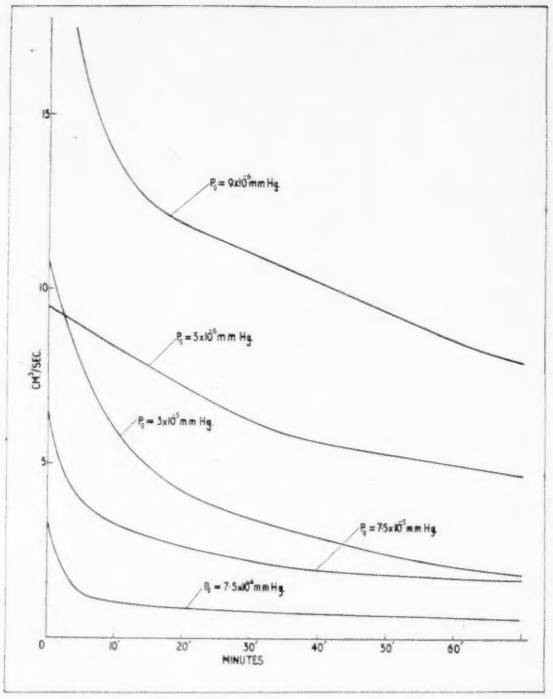


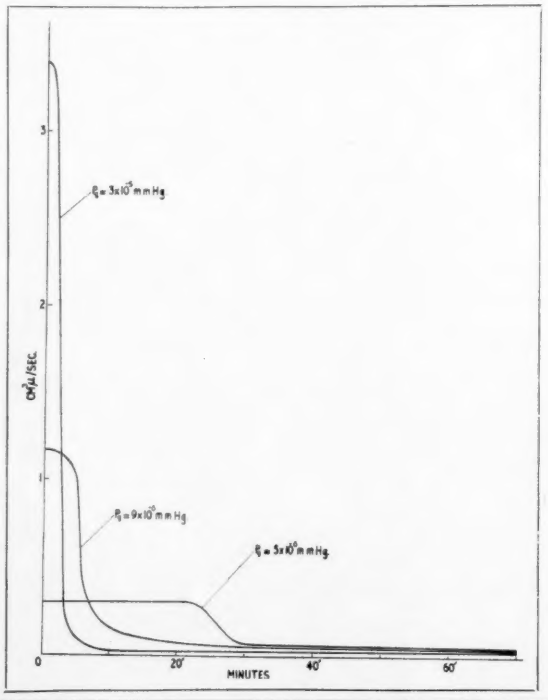
Figs 9a and 9b (above). Hydrogen.

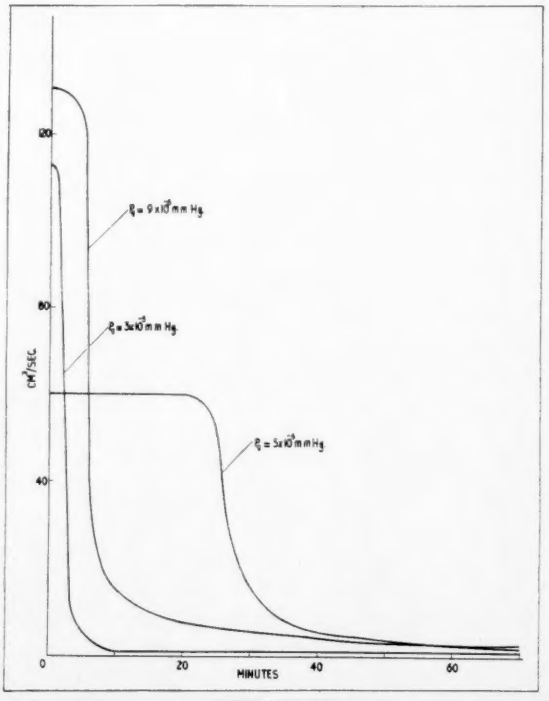
Figs. 10a. and 10b (below). Carbon dioxide.

Values of the Gettering Rate

Obtained at Various Pressures with a Barium Mirror of 1 cm.²

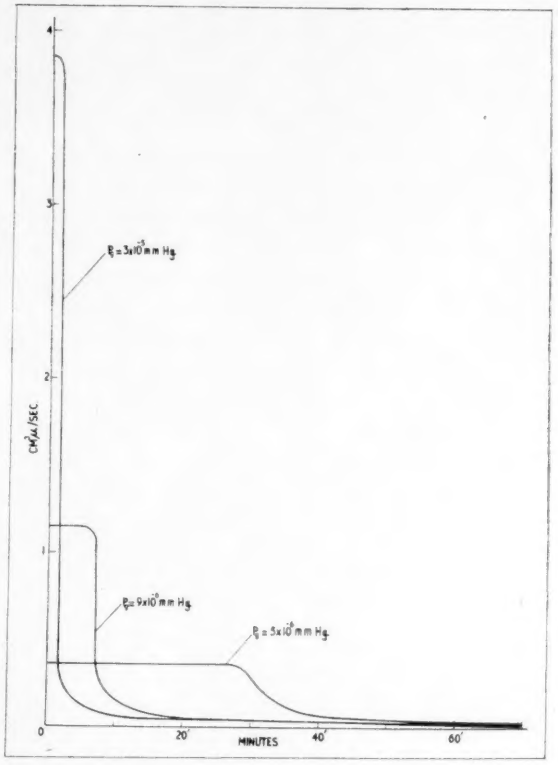






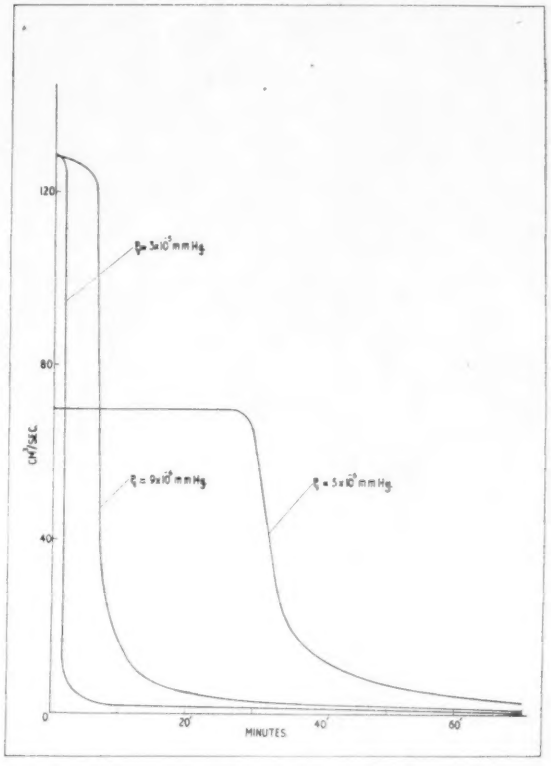
Values of the Instantaneous Capacity

Obtained at Various Pressures with a Barium Mirror of 1 cm.²



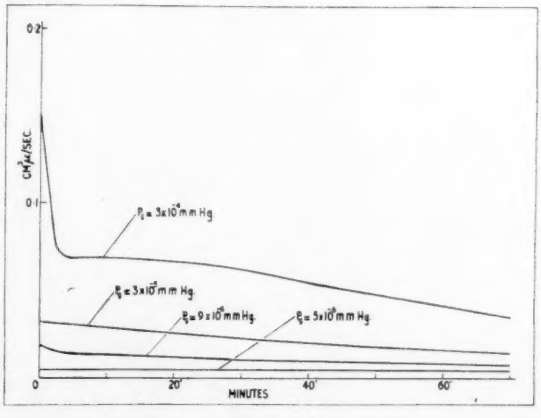
Values of the Gettering Rate

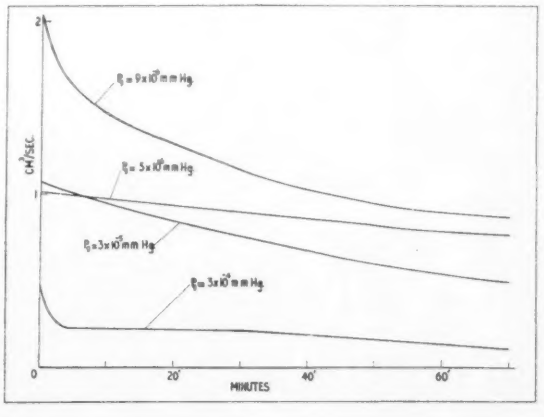
Obtained at Various Pressures with a Barium Mirror of 1 cm.²



Figs. 11a and 11b. Carbon monoxide.

Figs. 12a and 12b. Nitrogen.





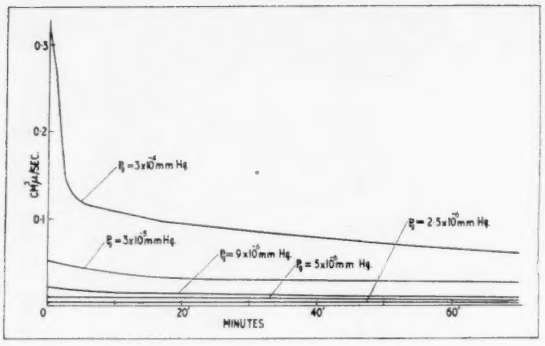
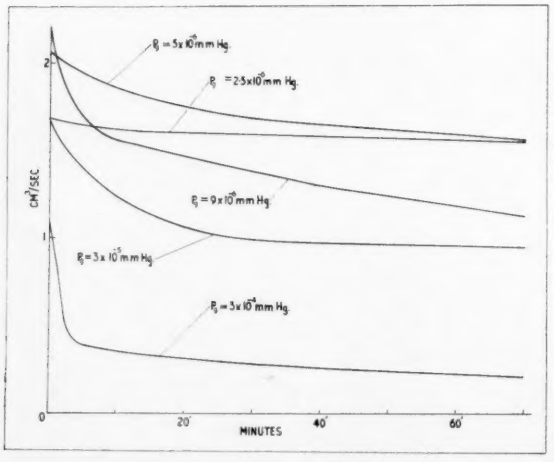


Fig. 13*a*. Values of the instantaneous capacity for air obtained at various pressures with a barium mirror of 1 cm.^{2} area.

hydrogen. Finally considering an earlier statement that the getter action with respect to hydrogen is affected by the presence of a discharge[7], it appears that the gettering of hydrogen shows rather anomalous characteristics requiring further exploration. Curves of instantaneous absorption capacity and gettering rate in different pressure ranges are shown in Figs. 9a and 9b.

Hydrogen is likely to be taken up in appreciable

Fig. 13*b*. Values of the gettering rate for air obtained at various pressures with a barium mirror of 1 cm.^{2} area.



amounts by the valve components during reduction treatment. It readily dissolves in metals but its liberation during the service life of the valve is slow and protracted. Hydrogen is not particularly dangerous to cathode emission but, as normally large quantities of it are present in the valve, the getter should be designed to deal with hydrogen just as efficiently as with other gases.

Carbon Dioxide and Carbon Monoxide. The total absorption capacities for carbon dioxide and carbon monoxide were determined at 0.6 and 0.72 litre micron per sq. cm. respectively. Figs. 10a and 10b, 11a and 11b show the curves of instantaneous absorption capacity and gettering rate for both gases at different pressures. Absorption of both gases is confined to the uppermost surface of the getter deposit. In the case of carbon dioxide the absorption product is probably Ba CO_3, one gramme molecule of which has a volume of 44.5 c.c. In both cases the absorption products form an impermeable layer on the getter surface which accounts for the previously mentioned abrupt cessation of gettering activity after a short while. The phenomenon of getter poisoning is confined to these two gases and the only known possible remedy is intense heating of the getter which may cause a certain amount of the gases to penetrate into the body of the deposit, thus partially restoring the effectiveness of the getter. Poisoning of the getter by these gases will account for many instances where valves are found in the production tests to have a poor vacuum, although the mirror deposit appears to be intact.

Carbon dioxide collects in the valve in large amounts during the activation of the cathode[15] and originates from the conversion of carbonates into oxides at the cathode. At this stage of valve production the getter is not yet evaporated and therefore the amount of gas in the valve after sealing-off depends exclusively on the efficiency of the pumps. Previous workers [5,15] estimated the partial pressure of carbon dioxide gas in the valve to be of the order of 10^{-6} mm.Hg, taking into account the known values for the dissociation pressure of carbonates. Penotet[16] states that the poisoning of the cathode by carbon dioxide is normally only of a temporary nature and emission recovers as the gas is gradually absorbed by the getter.

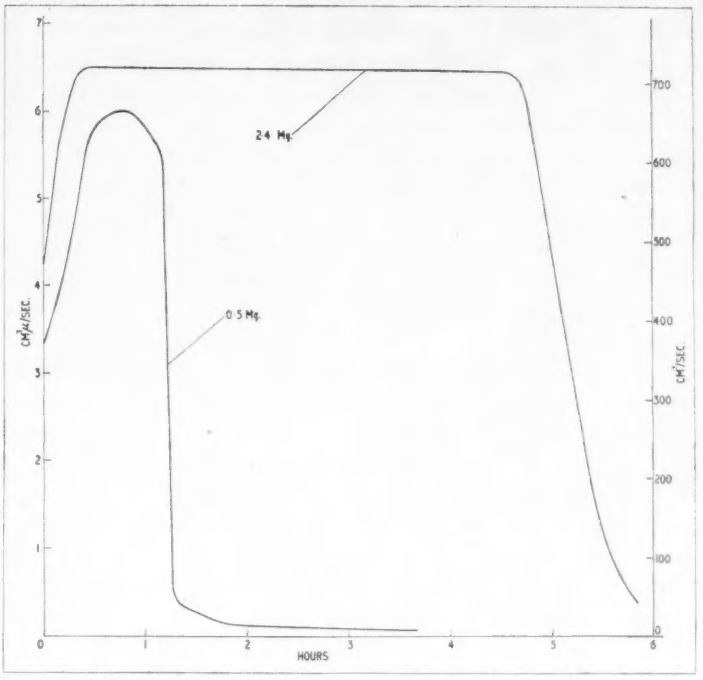
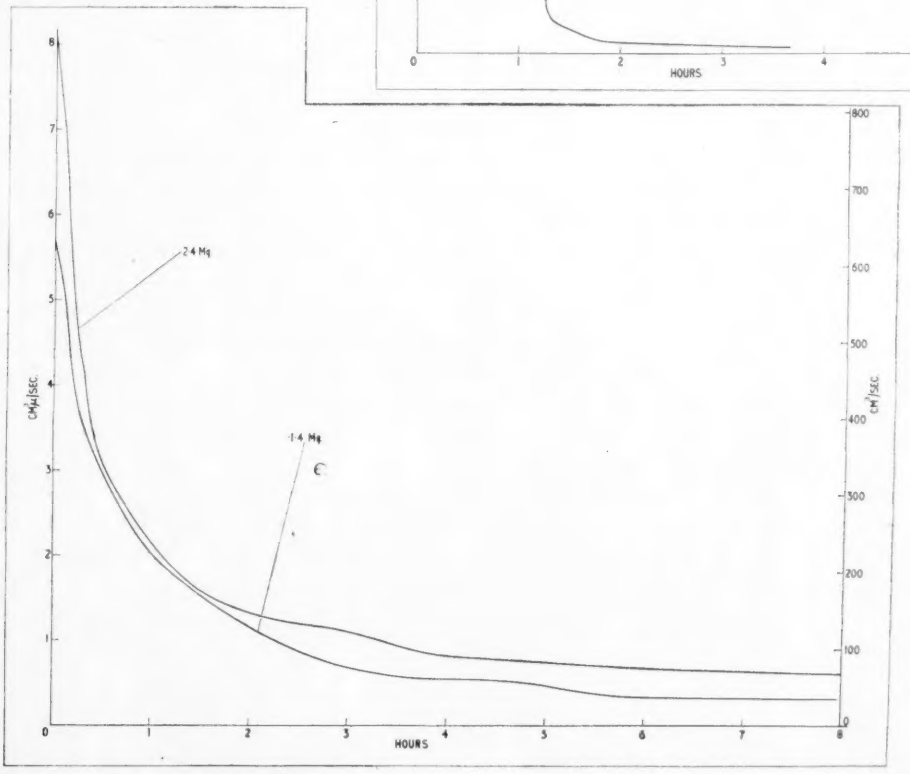


Fig. 14. Values of the instantaneous capacity and the gettering rate for oxygen, determined at p_g = constant = 9×10^{-6} mm.Hg, obtained with two barium deposits of equal area (8 cm.^{2}) but different weights.

Fig. 15. Values of the instantaneous capacity and the gettering rate for moisture, determined at p_g = constant = 9×10^{-6} mm.Hg, obtained with two barium deposits of equal area (8 cm.^{2}) but different weights.



Carbon monoxide, however, forms a large fraction of the residual gases in valves, an effect which was confirmed by Stoll[18] and other workers. This is why the problem of the poisoning of the getter by these gases is one of exceptional importance.

Nitrogen. The average total absorption capacity for this gas was determined at 0.33 litre micron per sq. cm. The values obtained in the experiments differ and this is attributed to the fact that the composition of the absorption product is variable. $Ba_3 N_2$ may be formed, one gramme molecule of which has a volume as small as 23 c.c. and will cause the mirror to become porous, or $Ba (N_3)_2$ is formed which has a volume of 76 c.c. This condition may be responsible for the phenomenon that the total absorption capacity of the deposit changes with the weight of the barium evaporated. The instantaneous capacity and gettering rate however depend on the surface areas of the deposits. For details of performance see Figs. 12a and 12b. Nitrogen does not seem to affect cathode emission appreciably and many workers treat it as an inert gas in this connection.

Air. The total absorption capacity of dry air was found to be 0.64 litre micron per sq. cm. Although the absorption process is confined to the uppermost part of the surface of the deposit it appears that the weight of barium evaporated has a certain influence on the absorption capacity of the getter for air. (See Figs. 13a and 13b).

CONCLUSIONS

The investigation reported in this paper was restricted to the performance of pure barium mirror deposits produced by the evaporation of a barium-aluminium alloy.

We aimed at assessing getter performance under conditions encountered in practice. The method we employed in our experiments was the same as that employed by previous workers, *i.e.* the now almost traditional capillary method. It is known that this test can be operated keeping the pressure in the manifold constant or alternatively keeping the pressure in the getter chamber constant. Nearly all data so far known on gettering rates have been obtained, keeping $p_m =$ constant. The author found when employing initially the method $p_m =$ constant that the values obtained for the gettering rate bore no relation to the actual absorptive power of the getter. On the other hand by employing the alternative method of $p_g =$ constant the results obtained are independent of the limitations given by inherent design features of the test equipment and are generally applicable to all cases where pressure and temperature are the same as in the experiments.

From a practical point of view it is preferable to assess getter characteristics by their absorption capacity instead of by gettering rates as defined in the past. The merits of assessing getter performance in terms of 'instantaneous absorption capacity' defined in $cm.^3 \mu/sec.$ are discussed. This method of recording has the additional advantage that the total absorption capacity can be calculated by integrating the curve in the graph showing the values determined experimentally for the instantaneous absorption capacity.

Results are given for the getter performance with respect to a number of gases obtained by employing the revised method.

Finally, the outcome of our experiments appears to suggest that there is a maximum gettering rate which is reached when operating at a certain pressure level. This phenomenon, if established, would be of considerable interest and requires further exploration.

The author is indebted to Dr. Origlio, a member of this laboratory, for his invaluable assistance in the experiments which form the basis of this paper.

For references see opposite page.

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References

[1] KOBAYASHI, A. and FURUYA, S., *J. Phys. Soc. Japan*, **6**, (1951), 238-243.
'Reactions of Barium Films with Oxygen'.

[2] WAGENER, S., *Brit. J. Appl. Phys.*, **1**, (1950), 225-231.
'A Method for Measuring the Efficiency of Getters at Low Pressures'.

[3] WAGENER, S., *Brit. J. Appl. Phys.*, **2**, (1951), 132-138.
'Efficiency and Mechanism of Barium Getters at Low Pressures'.

[4] WAGENER, S., *Proc. I.E.E. Part III*, **99**, (1952), 135-147.
'The Production of Very High Vacua by the Use of Getters'.

[5] WAGENER, S., *Vacuum*, **3**, (1953), 11-23.
'The Use of Getters for the Production of Very High Vacua'.

[6] WAGENER, S., *Proc. Phys. Soc.*, **66B**, (1953), 400.
'Sorption of Gases at Very Low Pressures by Thorium Powder'.

[7] WAGENER, S., *Nature*, **173**, (1954), 684-685.
'Influence of Electronic Impact on the Rate of Sorption of Gases on to Getter Materials'.

[8] WAGENER, S., *Z. Angew Phys.*, **10**, (1954), 431.
'Die Herstellung von Höchstvacua mit Hilfe von Getterwerkstoffen'.

[9] DAHLKE, W., *Le Vide*, **10**, (1955), 318.
'Tubes à Longue Durée'.

[10] BAILLEUIL-LANGLAIS, J., *Le Vide*, **10**, (1955), 380.
'Influence et Mesure du Degré de Vide dans un Tube Electronique à Cathode à Oxydes'.

[11] DEB, S., *J. Brit. I.R.E.*, **25**, (1951), 157.
'Decay of Emission from an Oxide-Coated Cathode due to Absorption of Matter Liberated from the Anode'.

[12] HIBI, T., and ISHIKAWA, K., *J. Appl. Phys.*, **22**, (1951), 986-987.
'On the Initial Decay of Oxide Coated Cathodes'.

[13] DORGELO, *Rev. Tech. Philips*, **14**, (1953), 198-199.
'Tubes d'Emission de Grande Puissance à Cathode en Tungstène Thorie'.

[14] ESPE, W., KNOLL, M., and WILDER, M. P., *Electronics*, **23**, (1950), 80-86.
'Getter Materials for Electron Tubes'.

[15] JACOBS, H., and WOLK, B., *Proc. I.R.E.*, (November 1949), 1247-1251.
'Microanalysis of Gas in Cathodic Coating Assemblies'

[16] PENOTET, H., *Le Vide*, **10**, (1955), 362.
'Les Empoisonnements des Cathodes à Oxydes'.

[17] LEVERTON, W. F., and SHEPHERD, W. G., *J. Appl. Phys.*, **23**, (1952), 787-793.
'The Use of Radioactive Isotopes in a Study of Evaporation from Thermionic Cathodes'.

[18] STOLL, S., *Brit. J. Appl. Phys.*, **7**, (1956), 94.
'Origin and Analysis of Gas in Electronic Tubes'.

Other Useful References

DUSHMAN, S., *Scientific Foundations of Vacuum Technique*. (John Wiley & Sons, Inc., New York, 1949), 654.

EHRKE, L. F., and SLACK, C. M., *J. Appl. Phys.*, **11**, (1940), 129-137.
'Contributed Original Research'.

HAASE, *Z. Angew Phys.*, **4**, (1950), 188-191.
'Die Getterwirkung dünner Barium filme bei tiefen Drucken'.

ARIZUMI, T., and KOTANI, S., *J. Phys. Soc. Japan*, **7**, (1952), 152-166.
'Absorption of Carbon Monoxide by the Barium Getter'.
Part I. Experiments at Low Temperatures.
Part II. Theory of Physical Adsorption.
Part III. Lattice Imperfections.

PLUMLEE, R. H., and SMITH, L. P., *J, Appl. Phys.*, **21** (1950), 811-819.
'Mass Spectrometric Studies of Solids. I. Preliminary Study of Sublimation Characteristics of Oxide Cathode Materials'.

AMAKASU, K., IMAI, T., and ASANO, M., *Le Vide*, **10**, (1955), 366.
'Effondrement de l'Emission des Cathodes à Oxydes'.

IMAI, T., *Le Vide*, **10**, (1955), 384.
'Détérioration de la Cathode à Oxydes par Dégagement Gazeux sous l'Influence du Bombardment Eléctronique'.

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MANIPULATION OF RADIOACTIVE GASES in high vacuum apparatus

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Summary

THE PROPERTIES OF RADIOACTIVE ISOTOPIC GASES, in particular tritium, krypton-85 and xenon-133 are described and an estimation of the radioactivity to be expected is given. The equipment available in A.E.R.E. Harwell for handling these gases is described in great detail with particular reference to the provisions made for protection against health hazards. The plant is made entirely of glass, the safe thickness of which can be calculated. Details are given of the vacuum system employed. Special manipulations described in the article cover the production and purification of tritium, tritiated water preparation, the preparation of tritium-zirconium targets, and the purification of krypton-85 and xenon-133. Besides their uses as research tools, the three nuclides are being increasingly used in industry. Among the applications mentioned are the uses of tritium for stabilising the break-down voltage in thermionic valves, the advantages of krypton-85 when employed for the elimination of static charges and the uses of kyrpton-85 and xenon-133 for air-flow indication.

Sommaire

LES PROPRIÉTÉS DE GAZ ISOTOPIQUES RADIOACTIFS, en particulier le tritium, krypton-85 et le xénon-133 sont données, ainsi qu'une estimation de la radioactivité devant en résulter. Description est faite, en grand détail, de l'equipement disponible au A.E.R.E. d'Harwell pour manipuler ces gaz, se référant en particulier au système de protection contre les radiations. L'appareil est fait entièrement en verre, dont l'épaisseur peut être calculée afin d'être protective. Des détails sont donnés sur le système à vide employé. Les manipulations spéciales décrites dans cet article englobent la production et la purification du tritium, la préparation d'eau contenant du tritium, de cibles de tritium-zirconium, et la purification de krypton-85 et de xenon-133. En dehors de leur emploi comme outils de recherche, ces trois produits sont de plus en plus utilisés dans l'industrie. Parmi les applications mentionnées, on note l'emploi du tritium comme stabilisateur de la tension de rupture dans les valves thermioniques, les avantages du krypton-85 employé pour éliminer les charges statiques et le krypton-85 et le xenon-133 comme indicateur d'écoulement d'air.

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INTRODUCTION

THE MANIPULATION of radioactive gases, as far as it is effected under vacuum, is carried out in systems essentially made from glass. The persistent growth of high vacuum technology has greatly promoted the development of vacuum glass systems and today many of their components showing the required precision are available commercially at a reasonable price.

It is the purpose of this article to describe up-to-date methods of manipulating radioactive gases in high vacuum apparatus and modern techniques of purifying such gases. Some general information on this subject can be found in the text books quoted in the bibliography[1-4].

It will be seen from the contents of this article that in many respects the procedures adopted are conventional and, though certain special precautions are indispensible in view of the radiation hazards encountered, the methods of manipulation are intrinsically no more difficult than in the case of non-radioactive work.

NATURE OF THE RADIOACTIVE GASES CONSIDERED

Three radioactive gases, in addition to a number of

* MS. received April, 1956.

non-radioactive gases, are handled in this laboratory. In order to present these gases in true perspective, a few details of their radioactive characteristics, which govern the correct procedure of manipulation with respect to health hazards, will be given. The three gases are tritium, krypton–85 and xenon–133. Occasional handling of carbon–14, in the gaseous form of carbon dioxide, and argon–37, has been undertaken, but will not be discussed in this article.

Tritium

The half-life of this radioisotope is 12.46 years[5-7]. It is a pure β–particle emitter with maximum energy of 18.9 keV and average β–energy of 5.69 keV[8]. No other radioisotope emits such a low-energy β–particle. The energy is so low that glass of 1 mm. wall thickness provides complete protection. The approximate minimum thickness of glass required to completely screen the tritium β–rays can be calculated from a formula by Libby which is only applicable to low energy β–emitters[9]

$$R_{max} = \frac{E^{5/3}}{150}$$

Where R_{max} = Range in mg./cm.^{2}
E = Maximum energy of β—particle in keV.

This formula gives a minimum thickness of 0.01 mm. glass for β–particles of 18.9 keV energy.

One standard millilitre of pure tritium contains 2.58 curies of radioactivity. This figure is derived from the disintegration constant, which, by one definition, is the fraction of the total number of atoms comprising the isotope which disintegrates in unit time. It is related to the half-life as follows :

$$\lambda = \frac{\ln 2}{t_{\frac{1}{2}}} = \frac{0.693}{t_{\frac{1}{2}}}$$

The total number of curies per c.c. of gas is calculated from

$$\chi = \frac{\ln 2}{t_{\frac{1}{2}}} \cdot \frac{NA}{VC}$$

Where V = Volume in c.c. NTP occupied by 1 g. mol gas
= 2.24×10^4
A = Atomicity
= 2 for tritium
N = Arogadro's number
= 6.03×10^{23}
C = Number of disintegrations/sec from 1 curie
= 3.7×10^{10} disintegrations/sec.

In the production of tritium, the radioisotope is invariably associated with hydrogen from which it can be separated by thermal diffusion. The isotopes equilibrate according to the reaction,

$$H_2 + T_2 \rightleftharpoons 2HT$$

and at equilibrium, by the law of mass action,

$$\frac{[HT]^2}{[H_2]\ [T_2]} = K_e$$

From thermodynamic calculation, which has been confirmed by experiment, the value of K_e is 2.56 at room temperature[10,11]. The equivalent value for H_2 and D_2 is 4. As the temperature of the system is increased, K_e rises until at about 600°C it reaches the value of 4. Unlike a mixture of H_2 and D_2, which can remain as separate molecules without exchange for some considerable time, a mixture of H_2 and T_2 will attain complete equilibrium within a few days. Increase of temperature in either case, as, for example, by use of a hot wire or absorption on pyrophoric uranium, hastens the attainment of equilibrium.

It has been established that the mechanism of the thermal exchange is a chain process involving atom-molecular interactions[12-14]. The reaction is initiated by the tritium β–radiation, and over a range of conditions half-time values of 260–818 minutes have been obtained.

Tritium decays to stable helium–3

$$^3_1H \rightarrow {}^{\ }_{-1}e + {}^3_2He$$

Since the atomicity of helium equals 1, the decay results in an increase in volume. From the well known disintegration formula :

$$N_t = N_o e^{-\lambda t}$$

it can be shown that approximately 6% of the tritium will decay in one year, producing a volume of helium–3 equivalent to about 12% of the initial volume of tritium.

In most cases where radioactive substances are confined in a glass vessel, the energetic nature of the particles emitted is sufficient to produce distortion in the crystal structure of the glass, and as a result imparts a certain colouration to it. Radium solution retained in a silica ampoule will gradually change the silica to a deep shade of purple, a colour which will disappear if the glass is heated to near the softening temperature. Krypton–85 will slowly turn Pyrex into a dark shade of brown. But the β–particles emitted by both carbon–14 or tritium are too soft to accomplish this effect, even though their concentration may be high. The presence of such isotopes within glass can be made visible only by coating the inner glass walls with a phosphor such as zinc sulphide or zinc orthosilicate. Scintillation light sources of this kind are only visible in a dark room, the zinc sulphide emitting a blue to blue-violet light, and the silicate a green light. Depending on the surface area-to-volume ratio and on the amount and constitution of the tritium within the glass ampoule, photon emission can vary up to a maximum of about 8×10^{11} photons cm.$^{-2}$ sec.$^{-1}$ [15].

With tritium, therefore, no special shielding precautions are necessary, the thinnest of glass vessels being sufficient to retain the emitted particles. Conventional high vacuum equipment in glass or metal can be employed for manipulation of the isotope, and special precautions are necessary only to cover the case of sudden breakage or inadvertent release of gas through the vacuum pumps or during maintenance work on the system.

Tritium will exchange rapidly with the inactive isotopes, hydrogen or deuterium. All vessels, whether glass, metal or ceramic, contain hydrogen in compound form on their surfaces, either as hydroxyl molecules, grease, or water vapour adhering strongly in small capillaries. Vacuum technologists are familiar with the difficulty of obtaining really high vacua in a system because of the outgassing phenomena. In most cases, thorough baking of all system components is required but this is not always possible in the vicinity of valves and seals of certain types. Tritium present in any concentration quickly contaminates a vacuum system, so that special precautions are necessary when measuring trace quantities of the nuclide. Even if a glass system which has previously held tritium is evacuated for many hours, an appreciable amount of tritium adheres to the glass, probably as part of another molecule. On exposure of the glass to air, the isotope once more exchanges with hydrogen in water vapour, and thus may constitute an appreciable health hazard depending on the original tritium concentration in the system.

Thus the precautions to be adopted when handling large amounts of tritium are : An adequate fume hood with a fast extraction fan, outlets of all pumps to be directed into extraction ducts, and the exercise of care in the construction and maintenance of the vacuum system, especially with regard to the regreasing of stopcocks.

Extensive studies on the absorption, distribution and excretion of tritium in and by men and animals have been conducted in America. It was found that the body absorbs the greater part of tritium inspired in the form of tritiated water, but only a small portion, if the tritium present was in the form of the molecule HT[16]. Small amounts of the inspired HT were found to be oxidised to HTO, which was accordingly absorbed. Turn-over of tritium in the body fluids following exposure, was in keeping with the total water content of the body, indicating a biological half-life of 12 days. Generally the experiments showed that the body will tolerate an appreciably greater quantity of tritium inspired as HT than inspired in the form of HTO by virtue of the low absorption. Nevertheless it was found necessary to fix the maximum permissible level (m.p.l.) of tritium present in terms of HTO, whether the gas is present in that form or not. The actual value of m.p.l., governing the work at the Atomic Energy Research Establishment (A.E.R.E.), is 5×10^{-5} μc/c.c. inspired air, based on 0.3 rad per 40 hr./week exposure.*

Krypton-85

Though a number of radioactive isotopes of krypton can be artificially produced, krypton–85 with the

* The rad is now the accepted unit of absorbed dose, corresponding to 100 erg per gramme of absorbing material. For protection purposes the rad and the roentgen may be assumed to be numerically equivalent[17].

conveniently long half-life of 10.5 years is the only one of real importance for use in industry and in tracer work [18–21]. It emits both β– and γ–rays, though the latter occur with only 0.6% of the β–disintegrations and have an energy of 0.54 MeV[22]. For tracer work with krypton this γ–ray intensity is insignificant, but for scales above millicurie level adequate precautions are necessary. The β–particles have a maximum energy of 0.695 MeV.

Krypton–85 is produced in the fission of uranium, occurring as 0.24% of the total fission products. In addition to the isotope of 10 year half-life, there is also an isomer of 4.4 hours half-life, which decays to the 10.5 year isotope. During decay a β–particle with E_{max}=0.85 MeV and γ–rays of 0.31 and 0.15 MeV energy are emitted. As some time elapses before the krypton–85 is extracted and purified, the proportion of this isomer has decayed to a negligible value.

The decay scheme with appropriate energy levels can be represented as shown in Fig. 1.

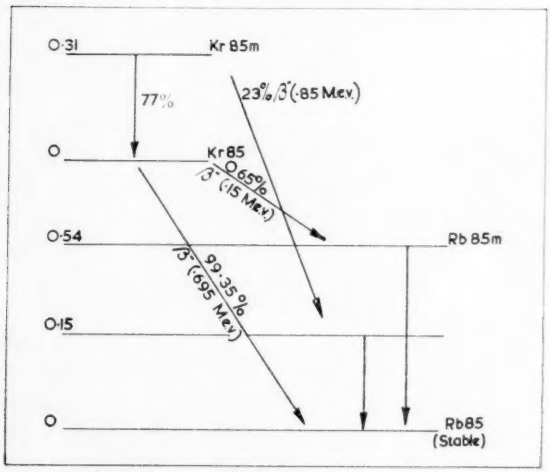

Fig. 1. Decay scheme for krypton-85

As in the calculations for tritium, 1 curie of pure krypton–85 will occupy a volume of 0.6 standard ml. In fission, however, it is associated with other stable krypton isotopes produced in the process. The abundance of krypton–85 is found to be about 7.5%, so that 1 st. ml. contains 0.12 curies. The following Table gives the abundance of isotopes in normal krypton and in fission krypton[23] :

Table I. Abundance of Isotopes in Normal Krypton and Fission Krypton

Normal Krypton		*Fission Krypton*	
Mass Number	*Relative Abundance*	*Mass Number*	*Relative Abundance*
78	0.35%	83	14.7%
80	2.27%	84	26.8%
82	11.5 %	85	7.5%
83	11.5 %	86	51.5%
84	57.0 %		
86	17.4 %		

The thickness of glass required to completely absorb the emitted β–rays, calculated from the formula

$$R = 0.543\, E_{max} - 0.133$$

where R = Range in mg./cm.^{2} aluminium
E_{max} = Maximum energy in MeV

can be shown to be 240 mg./cm.^{2} or about 1.2 mm. thick Pyrex glass. However, γ–rays are also emitted. Thus a $\frac{1}{4}$-inch thick lead sheet would be required to cut down the radiation tolerance of 1 curie to 1 m.p.l. at 20 cm. from the source.

The disintegration product of krypton–85 is rubidium–85, a stable isotope. In the course of one year the abundance of krypton–85 in fission krypton decreases from, say, 7% to 6.6%.

Unlike the conditions in the case of tritium, there is little contamination of glass or metal apparatus. But absorption of the gas by the stopcock grease can cause trouble in tracer measurements. Since this isotope is a rare gas, it cannot exchange with other isotopes present in compounds within the human system. Consequently the health hazard, through inhalation, is restricted to the effect of the β– and γ–rays during their rapid passage through the lungs. It is difficult for medical radiologists to establish a maximum permissible tolerance for krypton–85 in air, but it is probable that a shock dose of 10 mc. can be tolerated.

The precautions adopted are the same as in the

case of tritium, but no masks are used. When dealing with quantities greater than about 10 mc., lead shielding is employed.

Xenon-133

This radioisotope, too, is produced in fission of uranium, but trace quantities of low specific activity can be produced conveniently by a (n, γ) reaction in the pile on inactive xenon contained in a sealed silica ampoule. The half-life is 5.27 days and β– and γ–rays are emitted of 0.34 MeV and 0.081 MeV, respectively.

As with krypton–85, an isomer of xenon–133 is also produced during the fission process and since the half–life of this isomer is 2.7 days, it will naturally contribute to the total emission. By internal transition xenon–133m decays to xenon–133 emitting a γ–ray of 0.233 MeV. The abundance of xenon–133m is 1–2% of the xenon–133 yield[24]. In addition an isomer of xenon–131, with a half-life of 12 days, is obtained during the fission to the extent of 0.7% of the total amount of xenon–133. This again is internally converted to stable xenon–131 by emitting γ–rays of an energy of 0.163 and 0.08 MeV. The decay schemes and appropriate energy levels are shown in Fig. 2.

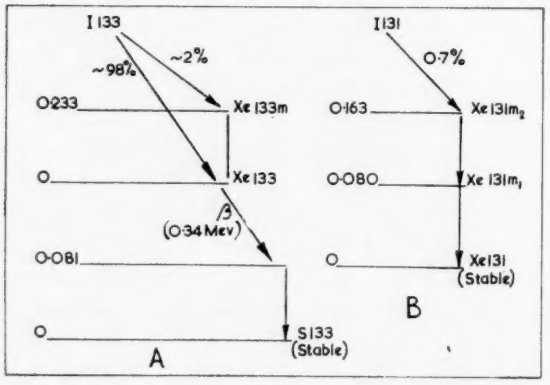

Fig. 2. Decay scheme for xenon-133.

To achieve the maximum yield by irradiation of a uranium slug requires a period of irradiation equal to ten times the half-life. Stable isotopes of xenon also occur in fission through disintegration of other products and the longer the irradiation, the lower the specific activity becomes. For 14 days irradiation, at a flux of 10^{12}ns/cm.^{2}/sec., followed by a decay lasting 2 days, the specific activity is about 120 curies per c.c. A 1 kg slug irradiated for 14 days at 10^{12}ns/cm.^{2}/sec. yields about 12 curies xenon–133 after a decay of 2 days.

The abundance of isotopes in normal xenon and fission xenon are shown in Table II.

Table II. Abundance of Isotopes in Normal Xenon and Fission Xenon

Normal Xenon		*Fission Xenon*	
Mass Number	*Relative Abundance*	*Mass Number*	*Relative Abundance*
124	.096%	131	13.4%
126	.090%	132	20.1%
128	1.92 %	134	35.8%
129	26.4 %	136	30.8%
130	4.07 %		
131	21.2 %		
132	26.9 %		
134	10.4 %		
136	8.9 %		

Table II does not include xenon–133, which, of course, will vary according to the time of irradiation and decay.

Manipulation of curie quantities of this isotope requires about $\frac{1}{4}$ inch lead shielding to provide complete protection. Again, vacuum apparatus made of glass is normally used, situated in a fume cupboard. The usual precautions for glass-blowing are adopted and exhausts of all pumps are directed into the extraction ducting. Being an inert gas, the same as krypton–85, its true toxicity is unknown, but no doubt a dose of 10 mc. can be tolerated in the body.

LABORATORY LAYOUT

The radiation activities involved in the work carried out in the laboratory to be described below are of the multi-curie level, and most of the equipment is constructed from glass. For these reasons it was considered advisable when designing the labor-

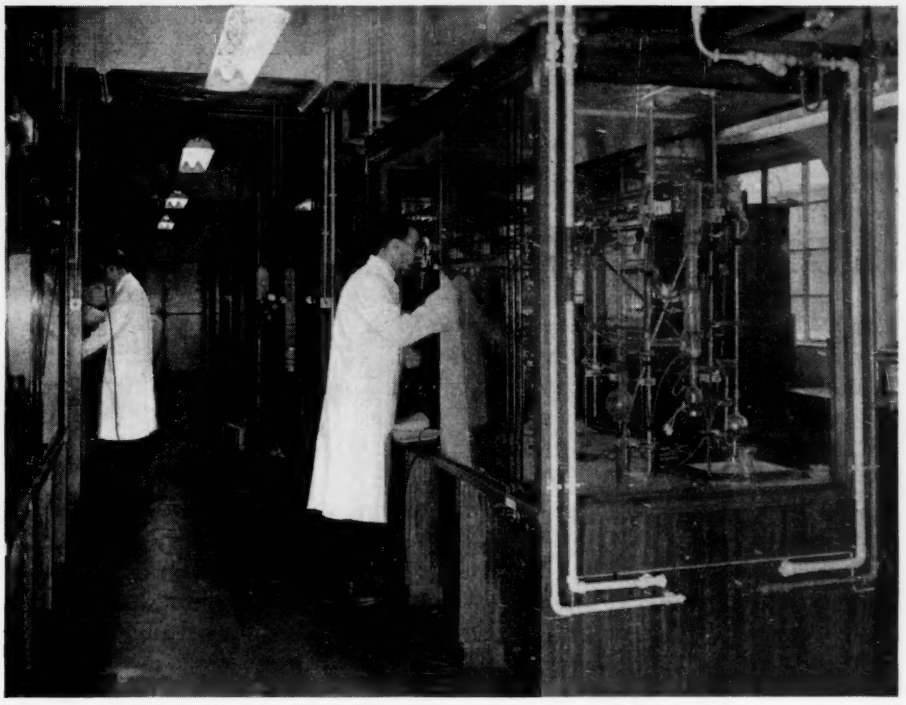

Fig. 3. Radioactive gas handling laboratory (A.E.R.E.)

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atory to arrange for the fume cupboards to be installed in individual units rather than in the form of, say, two long units extending over the full length of the laboratory. In the event of escape of active material inside one cupboard, the latter could be quickly and simply isolated without in any way interfering with the work in other cupboards. This is especially important in the case of isotopes of hydrogen which diffuse rapidly in air.

Fig. 3 shows the eight fume cupboards situated in the ' active ' area of the laboratory. The space available between any two cupboards is sufficient to allow for the installation of electronic equipment, or gas storage cupboards. The fume cupboards are connected by ducts to the main fan extraction system, each individual duct being provided with a damper to facilitate isolation if necessary from the main system. The latter is situated outside the laboratory and consists of two fans drawing the air from the laboratory to a chimney, 30 ft. high. Each fan is capable of extracting the air at a rate of 250 linear feet per minute. If both fans are working, the extraction rate is 400 linear feet per minute. During normal working conditions, one fan only is used and the other is brought into operation in the event of an accidental release of radioactive gases.

Each fume cupboard is 7 ft. 6 inch. high, 9 ft. long and 4 ft. wide. The working floor of the cupboard is 2 ft. 6 inch. above ground level. This height was chosen so that all parts of equipment are within easy reach in addition to providing adequate storage space within the cupboards. The vacuum rotary pumps are housed here together with ancillary electrical equipment which requires no attention apart from the normal regular maintenance. All pumps are supported on anti-vibration mountings making them barely audible when the doors of the cupboards are closed.

The working floor of each cupboard consists of 1–inch thick wood, rigidly supported and covered with ½–inch thick fire-proof material known as ' Sindanyo '. These floors can withstand the weight of the lead

screens required for the manipulation of krypton–85 and xenon–133. The sliding doors are of armoured glass. In an older cupboard design vertically raised glass windows were employed; this necessitated the provision of substantial supporting pillars which in many respects obstructed manipulation within the cupboard, especially during glass blowing operations. The sliding-door type of screen provides 6 ft. of clear space for manipulation but this is still not enough to allow two people to operate on one side of the cupboard at the same time. However, this is not a serious disadvantage.

Three 12-inch diameter holes in the roof of each cupboard provide the outlets to the extraction ducting. Connection to the main ducting is made with a rubber sleeve, to ensure that no vibration occurring in one cupboard is transmitted to another cupboard *via* the metal ducting. Fluorescent lighting tubes are fitted in recesses in the roof of each cupboard.

End windows, too, are of armoured glass, but in the case of three of our cupboards, this has been replaced by Perspex which facilitates the provision of glove ports for certain types of handling operations.

Services to the cupboard include water, gas, compressed air, electricity and oxygen. Three double watercocks are situated along the centre of the cupboard, which can be operated from either side. Gas, compressed air and oxygen valves are below the working floor, in a position not to cause an obstruction yet easily accessible. Electric power is supplied *via* 4 panels attached to the cupboard doors. Each panel carries one 15A and three 5A points.

One of the most essential items of the equipment for radioactive gas manipulation is, of course, the monitor. Since the energy of the β–particle emitted by tritium is very low, no normal type of β– and γ-monitor will suffice. The presence of tritium in the atmosphere can only be traced by a 'flow-through' ionisation chamber[25,26]. An economical way of passing air through the chamber is by using compressed air on the Venturi-pump principle. The air is blown across the opening to the chamber, resulting in air being drawn through at a constant rate. The monitor is fitted to the ceiling of the fume cupboard (see Fig. 4) and the electrometer and recorder are situated outside on a rack close by. The response of these instruments to a sudden presence of tritium in the atmosphere of the cupboard is rapid, in spite of the fact that the air has to pass through a long ion trap before it reaches the detection chamber.

As has been mentioned before, glass apparatus which has held tritium in gaseous form is liable to be highly contaminated. Therefore it is not possible to use the normal type of rubber blow tube with a mouth piece when carrying out glass-blowing work on such apparatus. Blowing by the mouth can be simulated by using compressed air, gently blowing through glass or metal T–pieces into the apparatus. The third arm of the T–piece is attached to the glass-blowing torch by a length of rubber tubing with an open end. A gentle squeeze of this tube will direct the compressed air to the hot work under construction and so provide the mild ' blow ' required. In the case of an apparatus which, prior to the glass-blowing operation, contained virtually pure tritium it is possible to create a tritium health hazard up to 1,000 m.p.l. before a break is sealed or an ampoule is correctly fixed to the apparatus. But the fast air extraction plant keeps the tolerance level in the region of the operator down to 2–3 m.p.l. during this manipulation. Even so, to prevent any health hazard whatsoever, a compressed air mask is worn during such work.

The remainder of the laboratory contains a series of storage cupboards for radioactive material and normal laboratory equipment. The former are wooden structures with Perspex doors connected directly to the extraction ducting. Thus, in the event of a storage ampoule containing tritium inadvertently cracking, the radioactive gases released will not create a health hazard in the cupboard. A number of fire appliances are provided, including buckets filled with calcium fluoride for use as a smothering agent in the event of a uranium fire.

VACUUM SYSTEMS

All mechanical pumps employed are of the 2–stage type, giving an ultimate vacuum of 5×10^{-4} mm. Hg and having a displacement of 22 l./min., see Fig. 5. Exhausts are connected through p.v.c. tubing to the

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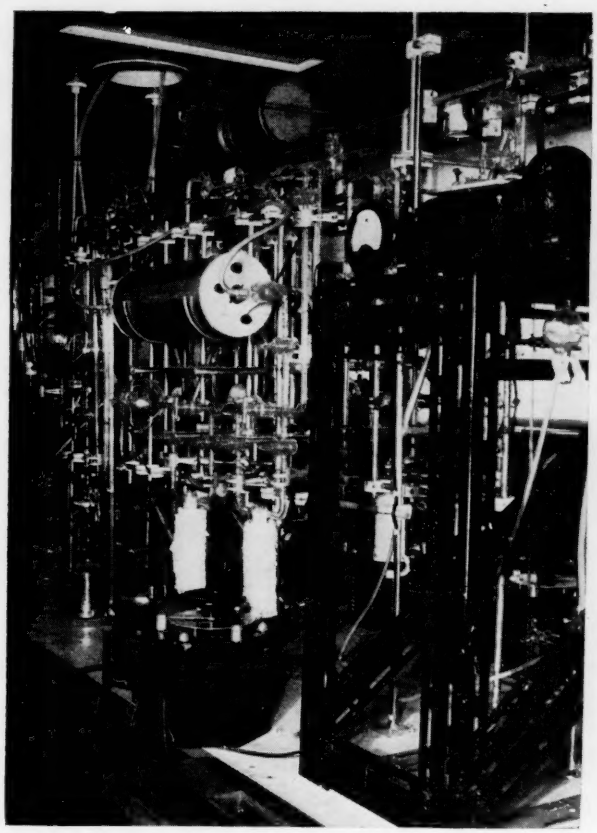

Fig. 4. Tritium preparation apparatus showing tritium monitor attached to ceiling of fume cupboard.

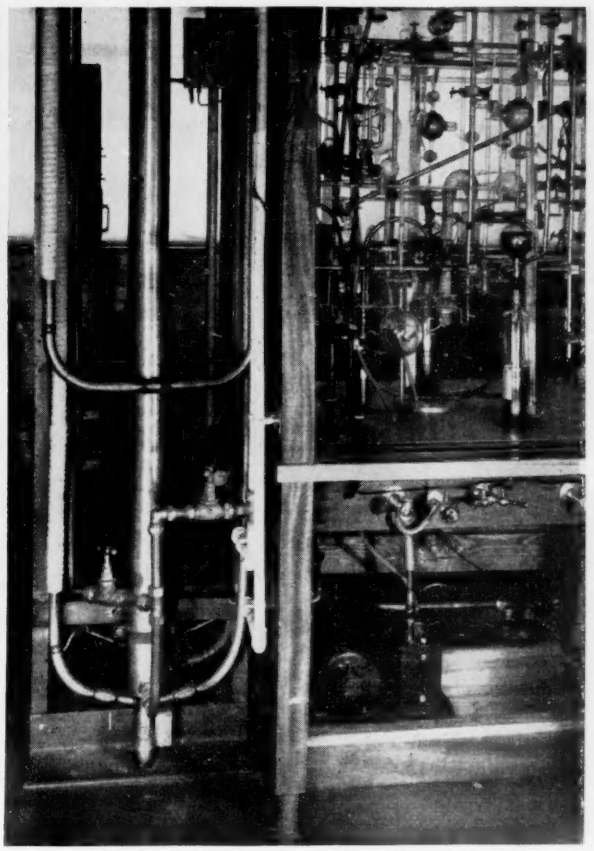

Fig. 5. Base of tritium thermal diffusion column with associated glass high vacuum apparatus (showing rotary pump housing).

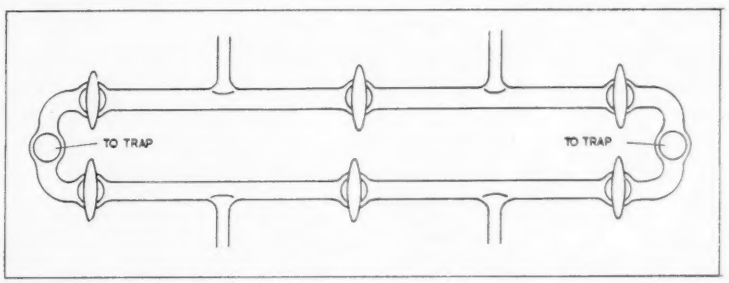

Fig. 6. "Birds-eye" view of a double-main vacuum manifold.

extraction ducting. In the working part of the cupboard there are two 2-stage mercury diffusion pumps constructed of Pyrex glass, with Dewar-type liquid air traps connected directly above each pump. The traps, one at either end of the cupboard, connect to a common manifold of 25 mm. internal diameter tubing, sections of which can be isolated by six 20 mm. bore high vacuum stopcocks as shown in Fig. 6. This arrangement facilitates the operation of four systems separately and simultaneously.

The frameworks supporting the glass vacuum systems are of $\frac{1}{2}$-inch mild steel sprayed with aluminium paint and screwed through the Sindanyo to the wooden base. There are additional supports from the ceiling of the cupboard.

No facilities are available for isolating the pumps in the event of the accidental release of radioactive gases to the main manifold. If a metal system were used, it would be essential to employ a magnetic valve in conjunction with an ionisation chamber in the pumping line but with a glass system the operational position of the stop-cocks is clearly visible and it is a comparatively simple expedient to establish a routine which ensures that all main manifold stopcocks are closed before introducing radioactive gases into the system. Likewise, since glass diffusion pumps operate fairly satisfactorily while the water in the cooling coil is warm, no automatic devices are employed to switch off the heater, should the water supply fail. A simple water-fountain tube situated adjacent to a red light which is in operation while the power is supplied to the heater, provides a simple indication that ' all is well '.

The remainder of the system is similar in many respects to the conventional vacuum apparatus, with a subsidiary vacuum manifold leading to a third mechanical pump for evacuating the reservoirs of McLeod gauges and Toepler pumps.

Special features include a hand-operated Toepler pump (see Fig. 7) which is used for measuring the small volumes of gas which are then compressed in the ampoule above. The mercury is raised as far as the constriction, the constriction is sealed and the ampoule is removed. This type of Toepler pump can also be calibrated and can then be used as a McLeod gauge.

Condensible gases are conveyed from one part of the vacuum system to another by an arrangement of charcoal traps and refrigerant traps but this arrangement is not satisfactory for the least condensible gases helium, neon, and hydrogen. With such gases, a Toepler pump is required, either the hand-operated type just mentioned, or more frequently in view of the large volumes manipulated in this laboratory, an automatic Toepler pump. A description of one design employed in this laboratory has been described elsewhere[27] and is shown in Fig. 8.

As the intensity of radioactivity involved in the work carried out in the laboratory increased, the design of the Toepler pumps was modified with a view to greater safety for the personnel and saving of material in the event of failure of certain parts. Though in the past it has been the practice to maintain a set maximum pressure in a system when circulating or collecting material, it is no longer possible to adhere to such precautions. It is obvious, therefore, that, should the magnetic valve leading to the vacuum pump fail to operate during the downward stroke of the Toepler pump, gas will be lost through the mercury reservoir, if the pressure in the system is greater than the depth of mercury in the reservoir. Such a failure could result in the loss of a few hundred curies of tritium but has never yet occurred during the four years of operation of Toepler pumps in this laboratory. Two precautions suggest themselves : (1) To lengthen the reservoir as shown in Fig. 9 which enables safe operation at a pressure up to 20 cm. Hg and (2) to introduce an additional sintered glass disc in the mercury reservoir which prevents mercury from moving further down the centre tube.

SPECIAL MANIPULATIONS

Tritium Production

In this country, tritium is produced by the pile irradiation of a lithium-magnesium alloy[28]. The thermal neutron irradiation of the lithium-6 isotope can be represented as follows :

$$^{6}_{3}Li + ^{1}_{0}n \rightarrow ^{4}_{2}He + ^{3}_{1}H$$

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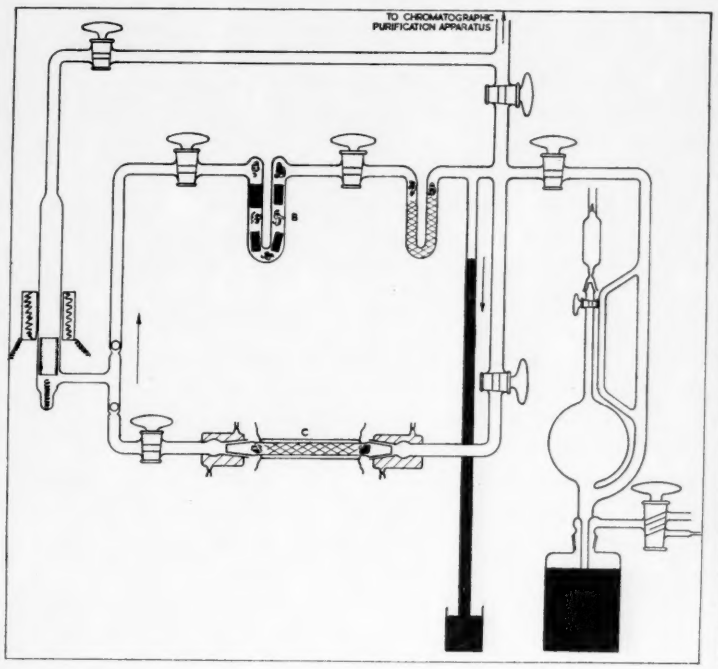


Fig. 7. Krypton-85 final purification apparatus.

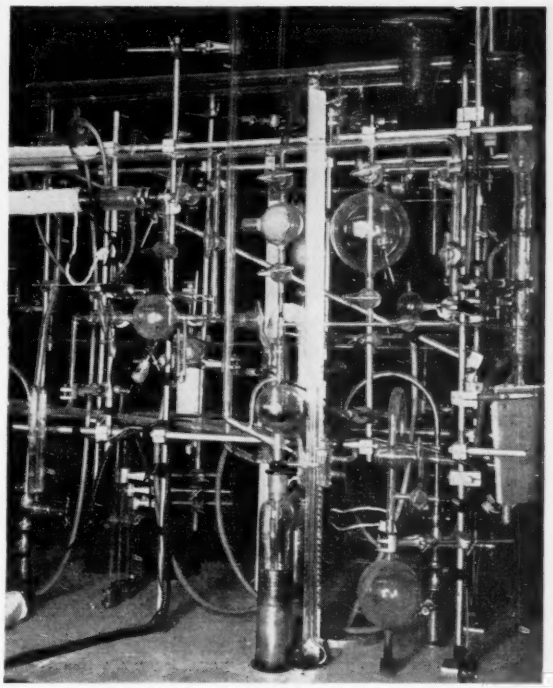


Fig. 8. Tritium manipulation apparatus showing one design of automatic Toepler pump.

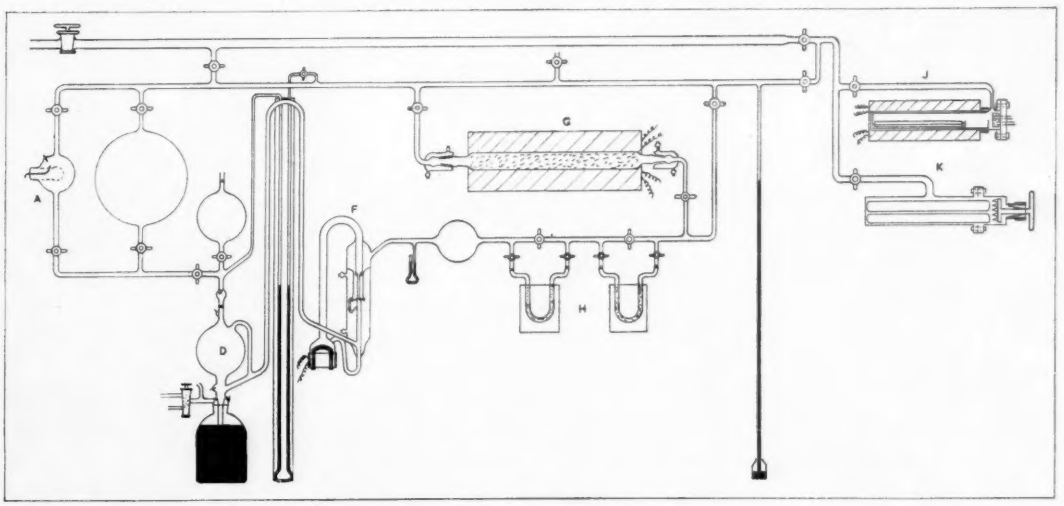


Fig. 9. Tritium preparation apparatus.

The abundance of the ${}^{6}_{3}Li$ isotope in natural lithium is 7.5% and the percentage of lithium in the alloy is 12%. Since the cross section of ${}^{7}_{3}Li$ to thermal neutrons is only 0.03×10^{-24} cm.^{2} (=0.03 barns), the yield of ${}^{8}_{3}Li$ which decays to ${}^{4}_{2}He$ is so low, that the latter contribution to the total ${}^{4}_{2}He$ produced is negligible (${}^{6}_{3}Li$ has a cross section of about 900 barns). Magnesium on irradiation results in ${}^{27}_{12}Mg$, a radioactive isotope with a short half-life. Consequently, providing no impurities of high cross-section are present within the target material, there is no γ-ray health hazard from irradiated rods.

The reactions which may occur during irradiation of this alloy are :

(1) ${}^{6}_{3}Li\ (n, \gamma)\ {}^{7}_{3}Li$	
(2) ${}^{6}_{3}Li\ (n, p)\ {}^{6}_{2}He$	$t_{\frac{1}{2}}$ 0.82 *secs.* $\xrightarrow[\beta]{}$ ${}^{6}_{3}Li$
(3) ${}^{6}_{3}Li\ (n, \alpha)\ {}^{3}_{1}H$	
(4) ${}^{7}_{3}Li\ (n, \gamma)\ {}^{8}_{3}Li$	$t_{\frac{1}{2}}$ 0.84 *secs.* $\xrightarrow[\beta]{}$ 2 ${}^{4}_{2}He$
(5) ${}^{7}_{3}Li\ (n, p)\ {}^{7}_{2}He$	
(6) ${}^{7}_{3}Li\ (n, \alpha)\ {}^{4}_{1}H$	

Reaction (4) has a neutron cross-section of only about 0.03 barn and the resulting helium contribution is less than 0.1% of the tritium contributed in Reaction (3). Reactions (5) and (6) do not occur. Also the (n,α) reactions with the three stable isotopes of magnesium are negligible. For practical purposes, Reaction (3) only need be considered. The same conditions apply where hydrogen (from protons) is contributed in the reactions listed above and in reactions with magnesium. Here again the total contribution is less than 0.1% of the total tritium produced.

The target material mentioned was chosen for the following reasons :—

(*a*) It has a high melting point (600°C) so that the rods are not materially affected by the irradiation.

(*b*) It is readily available and easily outgassed by baking *in vacuo* at 500°C to remove the dissolved hydrogen.

(*c*) The alloy does not attack the aluminium cans at the maximum temperature encountered during irradiation.

(*d*) Tritium recovery is effectively accomplished by re-heating the material to 500°C., releasing the gas by diffusion, but to effect complete recovery a melting and distillation procedure are required.

(*e*) The alloy can be re-used without further treatment. Storage in an inert and dry atmosphere, however, is necessary in order to avoid the absorption of hydrogen liberated through the action of the alloy on the water vapour in the air.

The alloy is irradiated in the form of rods of $\frac{1}{2}$-inch diameter. Because of the high neutron cross section of ${}^{6}_{3}Li$, however, the rods are self-shielding to neutrons to the extent of nearly 50%. The centre of the rod will, therefore, contribute very little to the production of tritium and if it were necesssary to obtain the maximum yield per unit weight of material, alloy in the form of a tube or as strips in a triangular or square formation would be necessary. Since the rods are re-irradiated, this consideration does not arise.

The tritium yield can be calculated from the well-known formula :

$$N_T = \frac{N_{Li}\sigma f}{\lambda_T}(1 - e^{-\lambda_T t})$$

where N_T = Number of tritium atoms present after irradiation for a time t sec.,
N_{Li} = Number of ${}^{6}_{3}Li$ atoms present in target material,
σ = Neutron cross section of ${}^{6}_{3}Li$ in cm.^{2},
f = Flux in neutron/cm.^{2}/sec.,
λ_T = Disintegration constant of tritium in sec.$^{-1}$.

Since there is 50% self-shielding in the case of $\frac{1}{2}$-inch rods of the alloy, the right hand side of the equation will have to be divided by 2.

The rods are hermetically sealed in aluminium cans with the air displaced by argon. After irradiation, the cans are punctured under vacuum (see Position K in Fig. 9) and the tritium which has partially diffused from the rods during irradiation is separated from the argon. No helium is obtained at this stage. The rods are then removed from the cans and baked under vacuum at a temperature up to 500°C. (J in Fig. 9),

as a result of which most of the tritium and at least 30% of the helium is released. The latter includes a few percent helium–3, the decay product of tritium. After completion of the extraction, the rods are ready to be used again. Only about 5–6% burn-up of $^{6}_{3}Li$ occurs during about a year of irradiation.

Purification of all evolved gases is effected in a silica tube containing uranium swarf (G) at 900°C., which forms solid compounds with all gases except hydrogen and the inert gases[29]. Any hydrocarbons, however, will decompose to hydrogen causing dilution of the tritium. Hydrogen isotopes are then removed from the gas stream by pyrophoric uranium (H). This is uranium in a very finely divided state prepared by the decomposition of uranium hydride and capable of absorbing hydrogen at room temperature. Since the vapour pressure of decomposition of uranium hydride does not exceed 10^{-5} mm. Hg at room temperature, hydrogen can be completely removed from the gaseous stream. The argon is pumped through the system by the Toepler pump (D), assisted by a mercury booster pump (F). Though ^{41}A and ^{37}A, radioactive isotopes of argon, occur during irradiation, their specific activity is too low and the demand for these isotopes not high enough to warrant saving them. Therefore after checking the volume of the argon and its residual activity, it is discarded. In the case of helium, however, the amount contributed is 1–3% $^{3}_{2}He$. This is large enough to warrant retention as a useful by-product either at this percentage or after suitable concentration in a thermal diffusion column.

Recovery of all tritium and associated hydrogen is complete when the pyrophoric uranium is heated to above 400°C. and the gases pumped off with the Toepler pump. The volume is measured and the percentage tritium in hydrogen calculated from the ionisation current registered in a glass ionisation chamber (A)[30]. The anode and cathode of this chamber are coated with a layer of conducting burnished silver and connected to a battery and electrometer. Currents ranging from 10^{-10}–10^{-3} amperes are measured. Calibration is made against mass spectrometric analysis with mixtures of tritium and hydrogen, where the atom percent tritium ranges from 10^{-4} to 90 or more. Although recombination of ions of the more concentrated gas occurs at high pressures in spite of applying up to 1,200 volt d.c., it is possible, using a spherical chamber of 200 c.c. capacity, to measure accurately all concentrations of tritium at pressures up to 30 cm. Hg. In addition to measuring the specific activity of the final product, the ionisation chamber serves as a means of checking residual tritium in the argon or helium.

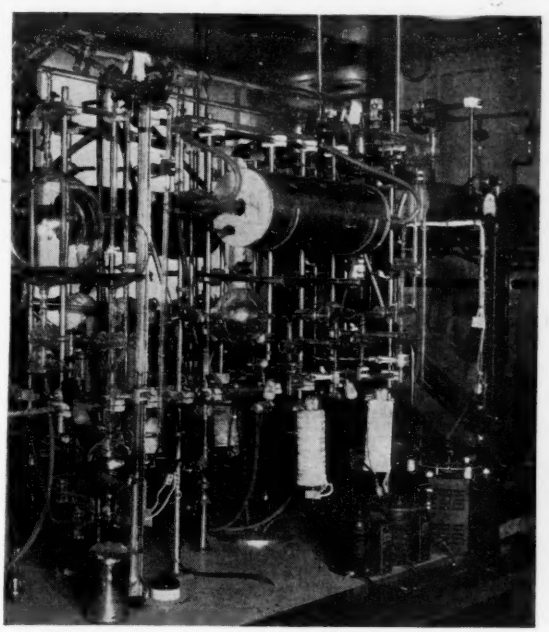


Fig. 10. Tritium preparation apparatus. See also *Fig.* 9.

Finally, the tritium is stored in a uranium bed, which is contained in a safe and transportable stainless steel tube equipped with a high vacuum metal valve. Details of the apparatus are given in Figs. 4 and 10.

Thermal Diffusion Separation of Tritium

It is very difficult to carry out all processes involved in the preparation and purification of tritium in an atmosphere completely free from hydrogen because any water vapour or any hydrocarbon present results in the liberation of hydrogen, and in order to eliminate hydrogen in the target material an extensive

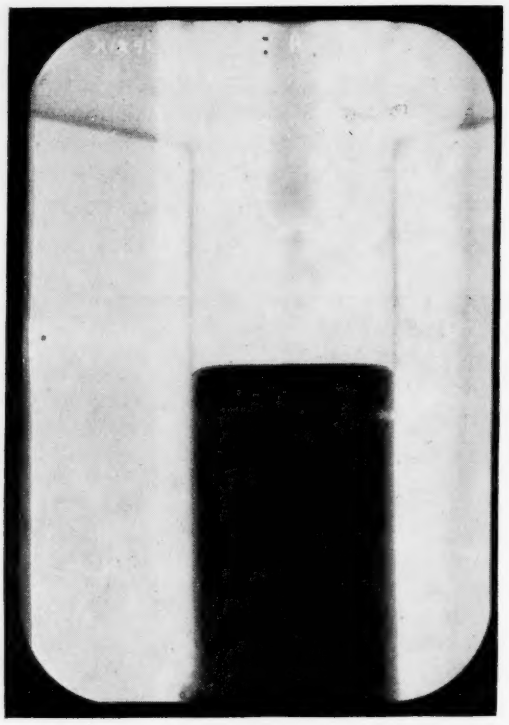


Fig. 11. Radiograph of mercury well of stainless steel thermal diffusion column.

process of re-melting under vacuum would be required. For this reason the final product is associated with a considerable percentage of hydrogen. For convenience and speed of separation, the thermal diffusion process is superior to all other methods of separating the isotopes of hydrogen[31,32]. A thermal diffusion column consists of a long tube of a suitable metal, down the centre of which extends a wire or narrow tube which can be heated electrically or chemically. The wall of the tube is maintained at room temperature by means of water. The thermal diffusion effect is such that there is a net transfer of momentum during molecular collisions from the heavier molecules to the lighter molecules of the gas. This net transfer of momentum depends upon the force law of interaction of the molecules in question, and in most cases it results in a partial pressure gradient of the lighter constituents parallel to the temperature gradient. The lighter molecules will, therefore, concentrate in the vicinity of the hot wire and be carried by the rising convection current to the top of the tube. In the same way, heavier molecules will accumulate by the cold wall and be carried downwards. There are two main effects which tend to counteract the thermal diffusion effect : (*a*) The remixing due to convection currents and (*b*) the remixing due to ordinary self-diffusion. At equilibrium, the sum of these two mixing processes just balances the transport by thermal diffusion.

We can represent the process by the formula[31]

$$\tau = H\psi(1-\psi) - (K_c + K_d)\frac{d\psi}{dz}$$

where τ = Net transport,
H = Transport coefficient,
K_c = Convection constant,
K_d = Self diffusion constant,
ψ = Concentration of the lighter (or heavier) constituent,
$\frac{d\psi}{dz}$ = The derivative of the concentration along the length of the column

At equilibrium, $\tau = 0$

$$\therefore H\psi(1-\psi) = (K_c + K_d)\frac{d\psi}{dz}$$

By integration

$$\frac{\psi}{1-\psi} = e^{2A(z-z_0)}$$

$$\text{where } 2A = \frac{H}{K_c + K_d}$$

If q_e represents the equilibrium separation factor, and is

$$q_e = \frac{C_B}{C_T}\frac{(1-C_T)}{(1-C_B)}$$

where C_B is the concentration of the lighter (or heavier) isotope at the bottom of the column and
C_T is the concentration of the lighter (or heavier) isotope at the top of the column.

Then it can be shown that

$$q_e = e^{2AL}$$

where L is the length of the column

$$\therefore \ln q_e = 2AL$$

For a particular design of column, the actual separation depends on the gas pressure. The variation of the separation factor with pressure can be represented by the Nier formula[33] :

$$\ln q_e = \frac{ap^2}{b + p^4}$$

where a and b are two constants which depend on the characteristics of the column and the gas.

Plotting $2AL$ against p we obtain a curve with a maximum value of $2AL$ occurring at $p^4 = b$. There is naturally no limit to the value of A which may be obtained, but constructional difficulties may arise and the narrower the column, the greater the pressure required to obtain maximum $2AL$. Because of the toxicity of tritium, it is wise to retain the maximum value below 1 atmosphere pressure in case of accidental leakage.

At Harwell, the column is constructed of stainless steel with a stainless steel centre wire electrically heated to about 800°C. The lower part of the column can be seen in Fig. 5. To allow for expansion the wire dips into a well of mercury at the bottom of the column. The total expansion of the wire gives a measure of the temperature. This can be measured by radiographing the bottom of each column, see Fig. 11. In addition special spacers are attached to the wire to maintain centrality. The net transport can be followed with ionisation chambers fitted at the top and bottom of the column.

Tritium concentrates at the bottom of the column and can be removed in small quantities, checked for purity and its concentration measured with external glass ionisation chambers of the design already mentioned. Abundances in excess of 90% can be achieved with starting material of low abundance.

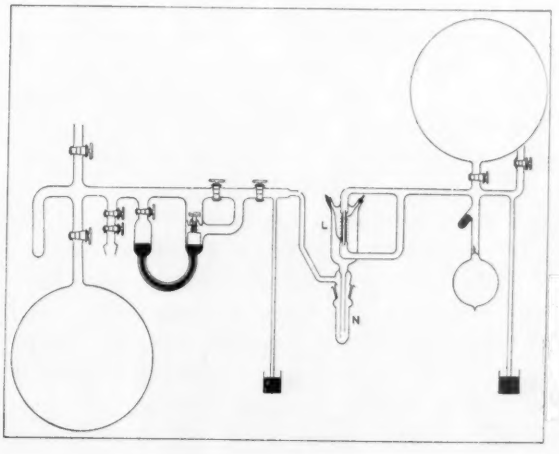
A thermal diffusion column is ideal for concentrating large volumes of tritium, but has the disadvantage of a large hold-up *i.e.* of a large amount of gas remaining 'stagnant' in the column. The higher the concentration required, the greater is the hold-up. Material, already of fairly high concentration, can be further concentrated even to as much as 99.9% by a Hertz pump method. This involves a series of vapour pumps capable of operating at backing pressures up to 20–30 mm. Hg[34].

Tritiated Water Preparation

The following method of preparing tritiated water has been adopted at Harwell. It yields a high purity product and minimises the risks of health hazards.

Of the possible ways of preparation, (*a*) hydrogen may be burnt in a current of oxygen using a heated platinum filament, but unless the partial pressure of oxygen is kept below 15–20 cm. Hg, there is a risk of explosion. (*b*) Alternatively, hydrogen is passed over hot copper oxide. This may result in certain copper salts being carried over into the refrigerant trap. (*c*) The Harwell method, depicted in Fig. 12, employs a palladium tube (L) which offers the two-fold advantage of a very pure product[35] and effective separation of the bulk of the hydrogen and oxygen. In operation the palladium is heated to about 200–300°C allowing only hydrogen to diffuse through. It contacts the oxygen on the outer surface of the palladium tube and is catalytically oxidised to water which is condensed in a cold trap (N) and removed from the apparatus. Virtually all the hydrogen (or tritium) diffuses through, and although only a few c.c. of tritium may be used in one run, it can be diluted with hydrogen, or followed by hydrogen, to provide an adequate

Fig. 12. Tritiated water preparation.



volume of tritiated water.

Because of the toxicity and gradual exchange of the tritium with moisture in the atmosphere, the product is maintained in a sealed ampoule, and only opened during dispensation.

Where the abundance of tritium in the water is high, the water decomposes at an appreciable rate as a result of radiation. The decomposition products, hydrogen and oxygen, can build up to high pressures within a few days. Pressures up to 600 cm. Hg[36] have been produced within 100 days by tritiated water containing 19% tritium. Since stored tritiated water at Harwell is maintained at 5 curie per ml. or less, the radiation decomposition is negligible.

Tritium—Zirconium Targets

Bombarding tritium with deuterons or protons results in a beam of neutrons of 14 MeV energy.

$$ {}_1^3H + {}_1^2H \rightarrow {}_2^4He + {}_0^1n $$
$$ {}_1^3H + {}_1^1H \rightarrow {}_2^3He + {}_0^1n $$

Such neutron beams used as bombarding particles can provide valuable data for physicists investigating nuclear reactions and fast neutron cross sections.

In order to obtain the strongest possible beam of monokinetic neutrons, it is essential to have the tritium distributed evenly over a thin layer of material. In addition, such targets must be stable under vacuum, adhere well to a suitable backing metal (which can be adequately cooled), and resist the strong local heating which occurs during particle bombardment. Tritium-zirconium targets prepared at Harwell produced by routine methods meet all these requirements.

Backing metals such as copper, silver and gold have been found suitable for the preparation of thin targets[37-41] covering the range of 0.2 to 1.0 mg zirconium per cm^2 of target surface. Discs cut from 0.005–0.01 inch sheet are ground with emery paper and soaked in trichlorethylene to remove any traces of grease, before being accurately weighed. In the evaporation apparatus a straight tungsten wire of 0.040 inch diameter is supported between two water-cooled copper electrodes. A specially designed water-cooled bell jar is placed over the electrode assembly and the apparatus evacuated to about 10^{-5} mm. Hg before the tungsten is outgassed at a temperature greater than 2,000°C. (see Figs. 13 and 14). The discs are then assembled in a hemispherical aluminium framework which is secured over the electrodes. About 100 mg of zirconium foil is crimped over the filament. The pressure in the chamber is again reduced to 10^{-5} mm. Hg, the zirconium is outgassed and then evaporated at about 2,000°C. Zirconium, being an ideal getter, will combine with the residual gases in the system but if the volume of the residual gases present is too large, the capacity of the zirconium to take up tritium will be considerably reduced. Hence, the provision of water-cooling for the electrodes and the bell-jar and the addition of an aluminium shield, which all contribute to reducing outgassing to a minimum. Within ten minutes the evaporation is complete, and when the power has been switched off, the pressure drops from a little above 10^{-5} mm. Hg down to $2–3 \times 10^{-6}$ mm. Hg as a result of the gettering power of zirconium.

No precautions are taken to prevent access of air to the targets, once sufficient time has elapsed for the targets to attain room temperature. A monolayer of oxide no doubt forms on the surface but this in no way influences absorption of tritium. After reweighing, the targets are placed, one at a time, in flat bottomed vessels which are attached to a Pyrex glass impregnation system (see Fig. 15). An evacuated glass tube is inserted in the vessel to reduce the gas space to a minimum.* Once the evaporation has been evacuated and leak tested, pure tritium is introduced from a pyrophoric uranium bed, to a pressure of 6–8 cm. Hg. The base of the vessel is heated to about 400°C and absorption of tritium is indicated by the rise of the mercury in the manometer. One target containing 1 mg of zirconium will absorb only 0.12 st. ml. tritium, hence the reason for restricting the capacity of the system.

A further check on the amount of tritium absorbed can be obtained by counting the bremsstrahlung emitted using an end-window Geiger-Mueller counter

* This apparatus was based on a design used by operators at Oak Ridge[43].

Fig. 13 (*above*)

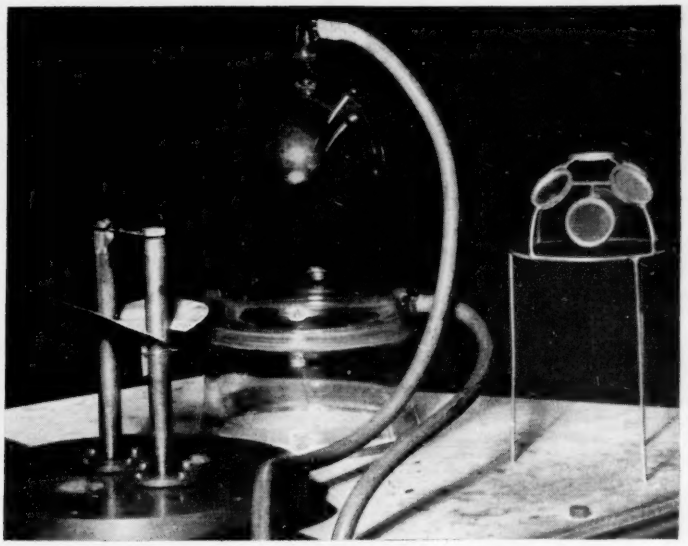


Fig. 14

Fig. 13. Zirconium evaporation apparatus (complete assembly).
Fig. 14. Zirconium evaporation apparatus (individual components).
Fig. 15. Tritium-zirconium target impregnation apparatus.
Fig. 16. Apparatus for thick tritium-zirconium target preparation.

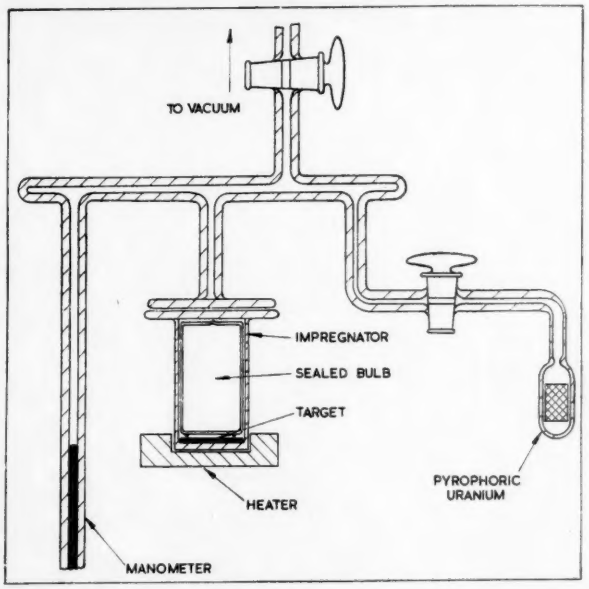
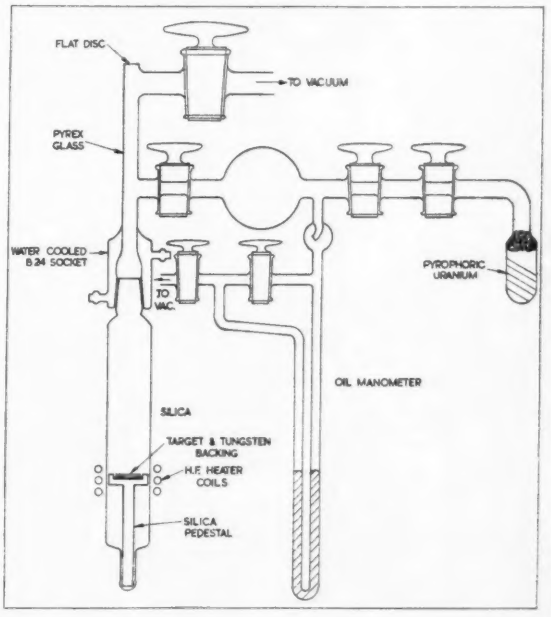


Fig. 15 (*below*)

Fig. 16



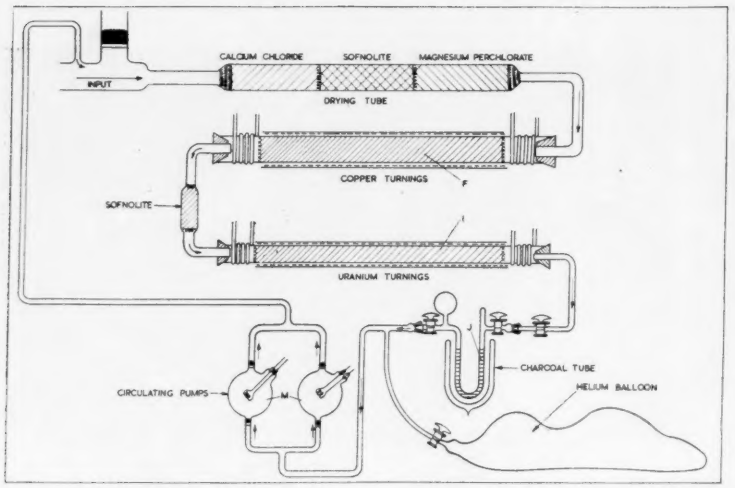


Fig. 17. Krypton-85 or xenon-133 initial purification apparatus.

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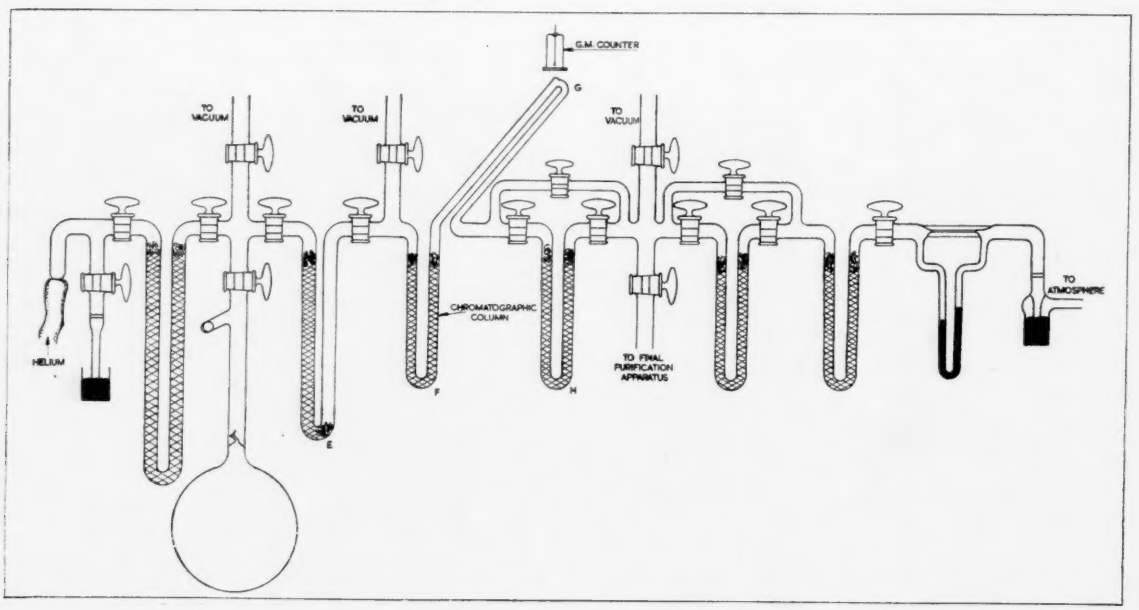
Fig. 18. Krypton-85 gas chromatographic apparatus.

situated about 3 cm. from the target[42].

In addition to thin targets, thick targets with zirconium layers of approximately 0.0005 to 0.002 inch thickness are prepared using tungsten as the backing metal[44]. These targets are prepared in a silica vessel where heat is supplied by an induction heater, both to outgas the tungsten and to melt the zirconium evenly over the surface of the tungsten (see Fig. 16). In all these targets, atomic ratios of tritium to zirconium vary between 1 : 1 and 1.5 : 1, and in the case of the thin targets no loss of tritium occurs when the targets are soft-soldered to a cooling device in the bombarding chamber of accelerating machines.

Preparation and Purification of Krypton-85 and Xenon-133

In the dissolution of uranium slugs, a fumeless dissolving method using nitric acid is adopted where



the nitrous fumes evolved are re-converted to nitric acid by pumping in oxygen[28]. The apparatus employed is shown in Fig. 17. Excess oxygen is removed by pumping it over heated copper turnings (F). In order to cope with the sudden burst of gas evolved by the dissolution, the gas collection apparatus is filled with helium at atmospheric pressure and is fitted with a balloon into which the extra gas can pass. This arrangement facilitates evening out of the gas bursts mentioned. Uranium swarf at 800°C. serves to remove any hydrocarbons present (I), activated charcoal (J) absorbs the rare gases and two circulating pumps (M), described in full elsewhere[27], maintain steady circulation through the purification train.

In the case of long irradiated slugs for krypton–85 production, the gases collected on the charcoal are separated by a chromatographic charcoal method (see Fig. 18). In this method the gases are re-adsorbed on charcoal (E), which is cooled with liquid nitrogen. The apparatus is then filled with helium to atmospheric pressure and the helium flows through the apparatus at a rate of about 3 l./h. On slowly removing the Dewar from (E) the gases are released and adsorbed at the head of the chromatographic charcoal column (F) which is maintained at −50°C. Then, due to elution with helium, argon is first removed, followed by krypton and finally xenon. The first collection charcoal (H) is by-passed during the initial phase, *i.e.* the argon discharge, but as soon as the emission of krypton is registered by the Geiger-Mueller end-window counter situated over a thinned-out portion of the glass-tubing (G), the flow is switched back to the first collection charcoal. Complete removal of krypton is indicated by the G.M.

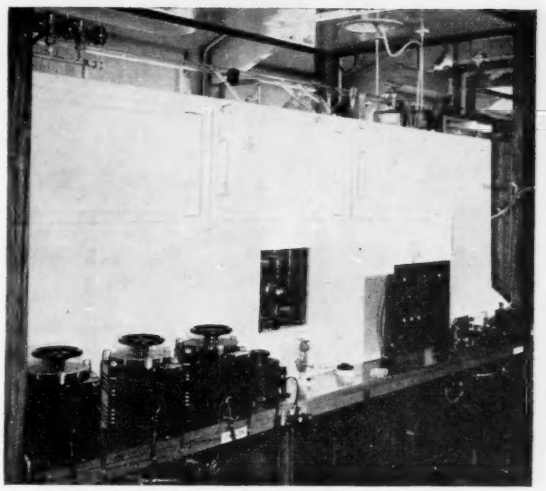


Fig. 19. Xenon-133 purification apparatus showing ¼ inch lead screen and lead glass window.

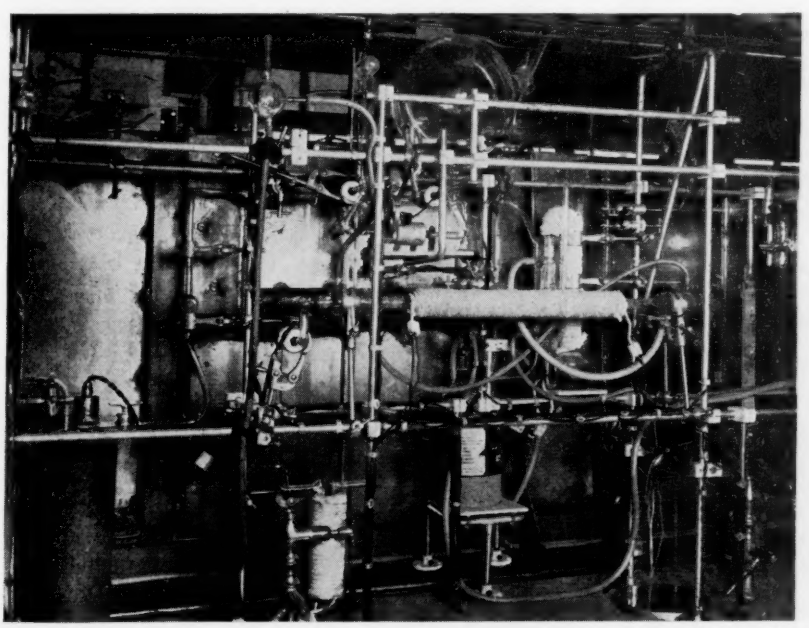


Fig. 20. Xenon-133 purification apparatus (behind the lead screen).

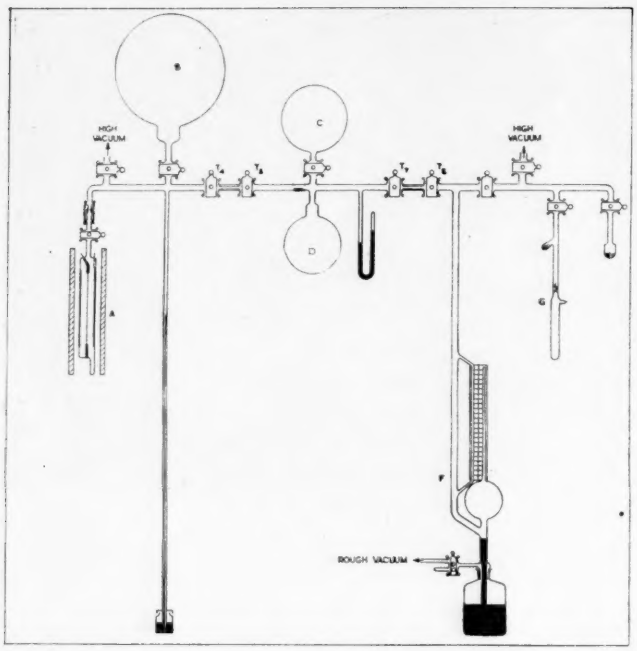
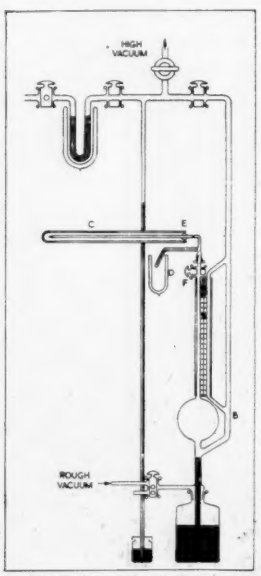


Fig. 21. Apparatus for assay of krypton-85 or xenon-133.

Fig. 22. Preparation of krypton-85 static eliminators.



counter returning to a near back-ground count.

Since certain hydrocarbons which may have escaped removal in the first purification apparatus, have adsorption coefficients similar to krypton, the krypton fraction is then circulated round a final purification apparatus (see Fig. 7), where the hydrocarbons are decomposed by hot uranium (C) and the resultant hydrogen absorbed by pyrophoric uranium (B).

Finally, the volume of krypton is measured (G) and the abundance of krypton–85 either measured mass-spectrometrically, or by an ionisation chamber.

In the production of xenon–133, uranium slugs need only be irradiated for a period of about 14 days to achieve practically the maximum yield. For such a short irradiation period, the quantity of krypton–85 produced is negligible. Dissolution and collection of the rare cases is by the same method as in the case of krypton–85, but final purification is conducted behind a $\frac{1}{4}$–inch lead screen within a fume cupboard (see Figs. 19 and 20). Solenoid valves capable of withstanding a pressure difference of more than 30 cm. Hg without leakage are employed instead of stopcocks to provide remote control manipulation. One or two conventional stopcocks are used, but need only be opened or closed in the absence of any radioactive material. Residual helium is first removed from the charcoal trap. The latter is then warmed up and the gases circulated over uranium to remove impurities. Any argon which may be present is removed simply by absorbing all the gases on the small charcoal bed at–80°C and opening this to a 5–litre bulb several times, with evacuation of the bulb after each expansion. The absorption of xenon on the charcoal is so appreciable and the volume so small that a negligible amount is lost during the process.

Finally the product is absorbed on a few mg of charcoal within a small capillary tube, which is subsequently sealed off. Such a source, which may be as high as 20 curies, is excellent for certain radiographical work. The gas may also be sealed off in certain types of ampoules equipped with septa, for

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use in lung experiments[45].

INDUSTRIAL USES OF THE NUCLIDES

There are extensive bibliographies published on the use of tritium as a tracer for medical, biological and chemical research[46,47]. Again, in the literature, extensive coverage is given to the various methods of assay of tritium, whether it be by ionisation chamber measurements, proportional counter, Geiger Mueller counter, scintillation counter or autoradiography; each method has its specific merit and means of reducing counter contamination to a minimum[48–58].

Assay of krypton–85 and xenon–133 can also be accomplished, and a diagram of a method employed at Harwell is illustrated in Fig. 21. This involves sampling out a small known volume of the original gas in (G) using the McLeod gauge (F). A small aliquot of this gas trapped between (T7) and (T8) is then expanded into (D) and diluted with inactive krypton or xenon from (C). A further small aliquot trapped between (T4) and (T5) is then expanded into the counter (A), and the appropriate gas filling (argon/alcohol) introduced from (B).

The use of all three radioactive gases is gradually extending into the industrial sphere where krypton–85 and tritium are beginning to supersede the use of other radioactive materials for the stabilisation of the break-down voltages of various gas discharge devices. The initiation of the discharge in these devices depends on the presence of free electrons. Otherwise, statistical time lags will occur even though the potential difference across the electrodes may exceed the normal break-down voltage. Many valves, operated in electronic equipment in a position where they are not exposed to light and not subject to the stabilising effect of ultra-violet radiation, will suffer from this defect unless they contain material which initiates the ionisation of the gas. Mainly daughter products of uranium have been used in the past, but krypton–85 has been suggested as a convenient source for this purpose, since it can be added without difficulty to the gaseous filling. Its handling too is easier and far safer than the radioactive materials previously used. Quantities of the order of 100 μc have been found to ensure satisfactory stabilisation[59].

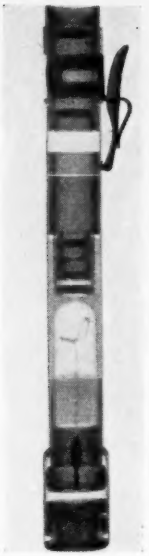

Fig. 23. Xenon-133 radiograph of a pocket ionisation gauge.

In certain cold cathode valves with gas fillings not exceeding a few cm. Hg, the low-energy β–particles of tritium have proved a very effective means of keeping a supply of free electrons in the region between the electrodes. The tritium may be part of the normal gas filling, or may be admitted for a period just long enough to contaminate the glass walls before it is removed. Sufficient gas is left behind to stabilise the break-down voltage of the valve[60].

Krypton–85 by its emitted β–radiation[61], can be just as effective as thallium–204 in the elimination of static charges. It can be used therefore with advantage as a filling gas in static eliminators of the kind made from commercially available aluminium tubes, 3 mm. in diameter and of a wall thickness of about 0.07 mm. After vacuum annealing at about 300–350°C., in order to remove greasy spots, the tubes can be effectively cold-welded by placing them between blunt-edged jaws and applying a pressure of 4,000 lb./sq. inch to the jaws. In the apparatus illustrated in Fig. 22, the aluminium tube, cold welded at one end, is ' araldited ' into narrow gauge

Pyrex glass tubing attached to a B–10 socket, above a hand-operated Toepler pump, (B). To protect the operator from the intense β–radiation, a thick-walled glass tube surrounds the aluminium tube (C). Sufficient krypton–85 to fill the tube to atmospheric pressure is absorbed on the small charcoal tube (D). Stopcock (F) is then closed and the gas in (D) transferred to (C) by expansion, after replacing the refrigerant by hot water. The constriction at (E) is sealed off, and a cold-weld on the aluminium tube very near (E) then seals the tube completely. Pure fission krypton filled at atmospheric pressure into such a tube will give a static eliminator containing about 6 mc per centimetre length and this compares favourably with the best available thallium–204 source. Apart from the long half-life, krypton–85 constitutes a particularly safe filling for static eliminators. If an eliminator is damaged mechanically the gas will be carried away by the ventilation system of the factory.

Other potential uses of radioactive gases in industry have been limited in the past to ventilation and leak detection experiments. Ventilation experiments have been conducted with xenon–133 and argon–41[62,63], so it can be assumed that krypton–85 will behave just as effectively. Tracer experiments to follow the flow of air in blast furnaces require the presence of an inert gas, and in the past radon has proved quite successful. However, because of the short half-life of this radioisotope, it is essential to co-ordinate experiments with the supply of the isotope. A difficulty such as this would be alleviated by using the long lived krypton–85. Gas leak detection from supposedly air-tight containers can be successfully undertaken using krypton–85. The minute amount of activity added to such vessels is too small to create a health hazard by virtue of the emitted γ–radiation. The long half-life allows the testing of vessels to be carried out over a period of years. The leaks are actually detected by means of a Geiger counter or an ionisation chamber.

Xenon–133 is not as useful industrially as the other nuclides because of its short half-life, but it has a high specific activity and a large adsorption coefficient on charcoal. It can thus serve as a high-activity point source in radiography. A typical radiograph is shown in Fig. 23, produced by a 1.7 curie source[64].

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References

1 DUSHMAN, S., *Scientific Foundations of Vacuum Techniques* (John Wiley & Sons Inc., New York, 1949).

2 FARKAS, A. and MELVILLE, H. W., *Experimental Methods in Gas Reactions* (Macmillan & Co. Ltd., London, 1939).

3 REIMANN, A. L., *Vacuum Technique* (Chapman & Hall Ltd., London, 1952).

4 GLASCOCK, R. G., *Isotopic Gas Analysis for Biochemists* (Academic Press Inc., New York).

5 JENKS, G. H., GHORMLEY, J. A. and SWEETON, F. H., *Phys. Rev.*, **75**, (1949), 701-702.

6 JENKS, G. H., SWEETON, F. H. and GHORMLEY, J. A., *Phys. Rev.*, **80**, (1950), 990-995.
'A Precise Determination of the Half-Life and Average Energy of Tritium Decay'.

7 JONES, W. M., *Phys. Rev.*, **100**, (1955), 124-125.
'The Half-Life of Tritium'.

8 HANNA, G. C. and PONTECORVO, B., *Phys. Rev.*, **75**, (1949), 983-984.
'The -Spectrum of Helium-3'.

9 LIBBY, W. F., *Anal. Chem.*, **20**, (1947), 2-6.
'Measurements of the Radioactive Tracers. Particularly C-14, S-35, T and Other Longer-Lived Low-Energy Activities'.

10 MATTRAW, H. C., PACHUCKI, C. F., and DORFMAN, L. M., *J. Chem. Phys.*, **20**, (1952), 926.
'Mass Spectrometric Determination of the Hydrogen-Tritium Equilibrium'.

11 UREY, H. C., *J. Chem. Soc.*, (1947), 562-581.
'Thermodynamic Properties of Isotopic Substances'.

12 FARKAS, A. and FARKAS, L., *Proc. Roy. Soc.*, **152A**, (1935), 124-151.
'Experiments on Heavy Hydrogen. V. The Elementary Reactions of Light and Heavy Hydrogen. Thermal Conversion of Ortho-Deuterium and the Interaction of Hydrogen and Deuterium'.

13 MEERSCHE, M. van, *Bull. Soc. Chim. Belg.*, **60**, (1951), 99-136.
'The Kinetics of Reactions Between Atomic and Molecular Hydrogen'.

[14] DORFMAN, L. M., and MATTRAW, H. C., *Knolls Atomic Power Laboratory*, A.E.C. Rep. KAPL 900, (1953). 'The Exchange Reactions of Hydrogen and Tritium'.

[15] ANON, *Tracerlog*, **69**, (1955), 3. 'Tritiated Light Sources'.

[16] PINSON, A., LA1218.

[17] ANON, *Brit. J. Radiol., Suppl. No.* 6, (1955). 'Report of the International Commission on Radiological Protection'.

[18] THODE, H. G., and GRAHAM, R. L., *Canad. J. Res.*, **25A,** (1947), 1-14. 'Mass Spectrometer Investigation of the Isotopes of Xenon and Krypton Resulting from the Fission of U-235 by Thermal Neutrons'.

[19] HOAGLAND, E. J. and SUGARMAN, N., *National Nuclear Energy Series, Div. IV*, **9,** Pt. 5 (McGraw Hill Book Co., Inc., New York, 1951), 635.

[20] WANLESS, R. K. and THODE, H. G., *Canad. J. Phys.*, **31,** (1953), 517-519. 'The Mass Spectrometer Determination of the Half-Life of Kr-85'.

[21] TURNER, J. F., *Atomic Energy Research Establishment, Harwell*, Report No. N/R 1254, (1953). 'The Half-Life of Long Lived Kr-85'.

[22] ZELDES, H., KETELLE, B. H. and BROSI, A. R., *Phys. Rev.*, **79,** (1950), 901. 'Radiation of Krypton–85'.

[23] ARROL, W. J., CHACKETT, K. F. and EPSTEIN, S., *Canad. J. Res.*, **27B,** (1949), 757-762. 'The Extraction and Purification of Xenon and Krypton Isotopes from Neutron Irradiated Uranium'.

[24] KATCOFF, S., and RUBINSON, W., *Phys. Rev.*, **91,** (1953), 1458-1469. 'Yield of Xe–133 in the Thermal Neutron Fission of U–235'.

[25] PITTENDRIGH, L. W. D., *Atomic Energy Research Establishment, Harwell*, Report No. HP/M/48, (1953). 'Continuous Flow Tritium in Air Monitor'.

[26] SHAW, D. F., *J. Sci. Instrum.*, **32,** (1955), 178-180. 'An Automatic Monitor for Measuring Tritium Contamination in Air'.

[27] WILSON, E. J., *Vacuum*, **3,** (1953), 287-288. 'An Automatic Toepler Pump and a Circulating Pump'.

[28] JOHNSTON, J. E., *et al. eds.* Radioisotope Conference 1954, **2,** (Butterworths Scientific Publications, London, 1954).

[29] STEDDING, F. H., *et al. Nucleonics*, **4,** (1949), 4-15. 'Uranium Hydride, I. Preparation Composition and Physical Properties'.

[30] WILSON, E. J., *Atomic Energy Research Establishment, Harwell*, Report No. I/M. 34, (1954). 'Ionisation Chamber Measurements with Tritium'.

[31] JONES, R. C. and FURRY, W. H., *Rev. Mod. Phys.*, **18,** (1946), 151-224. 'The Separation of Isotopes by Thermal Diffusion'.

[32] ALMQUIST, E., ALLEN, K. W. and SANDERS, J. H., *Rev. Sci. Inst.*, **26,** (1955), 649. 'Thermal Diffusion Column for Concentrating Tritium in Tritium-Hydrogen Mixtures'.

[33] NIER, A. O., *Phys. Rev.*, **57,** (1940), 30-34. 'The Concentration of Carbon-13 by Thermal Diffusion'.

[34] DUNN, F. J., MOSLEY, J. R. and POTTER, R. M., *Anal. Chem.*, **27,** (1955), 63-64. 'Separation of Mixtures of Tritium and Hydrogen using Hertz Pumps'.

[35] WILSON, E. J., *Atomic Energy Research Establishment, Harwell*, Report No. I/M. 28, (1953). 'The Preparation of Tritiated Water'.

[36] GHORMLEY, J. A. and ALLEN, A. O., *Oak Ridge National Laboratory, Tenn.*, Report No. ORNL. 128, (1948). 'Decomposition of Tritium Oxide under its own Radiation'.

[37] GRAVES, E. R., RODRIGUES, A. A., GOLDBLATT, M. and MEYER, D. I., *Rev. Sci. Instrum.*, **20,** (1949), 579-582. 'Preparation and Use of Tritium and Deuterium Targets'.

[38] LILLIE, A. B. and CONNER, J. P., *Rev. Sci. Instrum.*, **22,** (1951), 210. 'Preparation of Thin Tritium Targets'.

[39] ROCHLIN, R. S., *Rev. Sci. Instrum.*, **23,** (1952), 100-101. 'Preparation of Tritium-Zirconium Targets'.

[40] CONNER, J. P., BONNER, T. W. and SMITH, J. R., *Phys. Rev.*, **88,,** (1952), 468-473. 'A Study of the H-3 (d.n.) He-4 Reaction'.

[41] ARROL, W. J., WILSON, E. J. and EVANS, C., *Atomic Energy Research Establishment, Harwell*, Report No. I/R 1135, (1956). 'The Routine Production of Zirconium-Tritium and Zirconium-Deuterium Targets'.

[42] TASCHEK, R. F. and GITTINGS, H. T., *Phys. Rev.*, **74,** (1948), 1553-1554. 'Observations of Naphthalene Scintillations Caused by Tritium Beta-Rays'.

[43] MASSEY, B. J., Private Communication.

[44] EVANS, C., *Atomic Energy Research Establishment, Harwell*, Report No. I/M. 38, (1955). 'Preparation of Thick Zirconium-Deuterium Targets'.

[45] KNIPPING, H. W. *et al., Dtsch. Med. Wschr.*, **80,** (1955), 1146-1147. 'Eine Neue Methode zur Prefung der Herz-und Zungen Funktion, die Regionale Funktion Analyse in der Lungen-und Herz Klinik mit Hilfe des Radioaktiven Edelgases Xenon-133'.

[46] BROWN, W. G., KAPLAN, L., DYKEN, A. R. VAN, and WILZBACH, K. E., *International Conference on the Peaceful Uses of Atomic Energy (Geneva)*, 1955., Paper No. 149. 'Tritium as a Tool in Industrial and Chemical Research'.

[47] THOMPSON, R. C., *Nucleonics*, **12,** (1954), 31-35. 'Biological Applications of Tritium'.

[48] GLASCOCK, R. F., *Biochem. J.*, **52,** (1952), 699-704. 'Combustion Technique for the Assay of Tritium, Carbon-13 and Carbon-14, in a Single 10 mg Sample of Biological Material'.

[49] PAYNE, P. R., CAMPBELL, I. G. and WHITE, D. F., *Biochem. J.*, **50,** (1952), 500-502. 'Combustion of Tritium-Labelled Organic Compounds'.

[50] WING, J. and JOHNSTON, W. H., *Science*, **121**, (1955), 674-675.
'Method for Counting Tritium in Tritiated Water'.

[51] HAYES, F. N. and GOULD, R. G., *Science*, **117**, (1953), 480-482.
'Liquid Scintillation Counting of Tritium Labelled Water and Organic Compounds'.

[52] SWAIN, C. G., KREITER, V. P. and SHEPPARD, W. A., *Anal. Chem.*, **27**, (1955), 1157.
'Procedure for Routine Assay of Tritium in Water'.

[53] FINKELSTEIN, A. and LESIMPLE, M., *J. Nucl. Energy*, **2**, (1955), 101-109.
'Le Dosage du Tritium dans l'Eau Tritee'.

[54] EIDINOFF, M. L. and KNOLL, J. E., *Science*, **112**, (1950), 250-251.
'Measurement of Radioactive Hydrogen in Solid Samples—Comparison with Gas Counting'.

[55] FITZGERALD, P. J., EIDINOFF, M. L., KNOLL, J. E. and SIMMEL, E. G., *Science*, **114**, (1951) 494-498.
'Tritium in Radioautography'.

[56] FARMER, E. C. and BERSTEIN, I. A., *Science*, **117**, (1953), 279-280.
'Determination of Specific Activities of Tritium-Labelled Compounds with Liquid Scintillators'.

[57] GRENON, M. and VIALLARD, R., *J. Chim. Phys.*, **49**, (1952), 623-628.
'Determination of Tritium'.

[58] CAMERON, J. F., *Nature*, **176**, (1955), 1264.
'Measurement of Tritium in Water Samples'.

[59] JEFFERSON, S. and PUTMAN, J., *Brit. Pat.* No. 737, 386.

[60] ARROL, W. J. and JEFFERSON, S., *Brit. Pat.* No. 738, 182.

[61] ARROL, W. J., *Brit. Pat.* No. 698, 779.

[62] GRERON, J., *Radioisotopes Techniques*, II (Her Majesty's Stationery Office, London, (1953) 9.

[63] SELIGMAN, H., *Radioisotopes Techniques*, II (Her Majesty's Stationery Office, London, (1953), 9.

[64] MAYNEARD, M. V., *International Conference on the Peaceful Uses of Atomic Energy (Geneva)* 1955., **10** Paper No. 768.

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VACUUM IN RUSSIA

HIGH VACUUM EQUIPMENT*

Sommaire

COMPTE RENDU SUR l'état actuel de l'industrie du vide en Russie. Après une courte discussion sur les conditions de base requises pour un système à vide, et pour un équipment en général, des détails sont donnés sur la réalisation et les charactéristiques de différents types de pompes, de jauges de mesure, joints et appareils pour détecter les fuites, qui sont disponibles dans ce pays.

Introduction

During recent years, high vacuum techniques have been used on an ever widening scale in the field of nuclear energy, metallurgy, the food and chemical industry, and other branches of technology and science. In particular, the production of heat resisting steel, pure titanium and zirconium are based on the use of high vacuum plant and its present rate of production is closely linked with recent developments regarding this equipment. The manufacture and use of modern high vacuum equipment demand different methods and materials from those employed in general engineering. This springs from the exceptionally high requirements as to cleanliness of the internal surfaces and the quality of the seals incorporated in the apparatus. Experience shows that such equipment can only be built with success, if skilled and trained personnel are employed. In the United States and the United Kingdom, for example, the design and manufacture of high vacuum equipment and materials is concentrated in companies specialising in these activities. These companies supply the basic high vacuum components of apparatus for all kinds of applications, such as mechanical and diffusion pumps, fittings, leak detectors, and manometers, and carry out research on these components in their laboratories.

In addition, they develop high vacuum plant for special purposes, *e.g.* vacuum furnaces, vacuum stills, drying plant and apparatus for the preparation of thin metallic films, etc.

Industrial high vacuum equipment has to be designed to permit operation at measurable pressures ranging from several millimetres Hg to 10^{-5}–10^{-7} mm. Hg. The problem is not just that of pumping a receptacle down to a certain level of sub-atmospheric pressure, it is rather that of maintaining the enclosure at a given low pressure for a long time while certain processes are carried out in the receptacle which cause a considerable evolution of gas. The deciding factor, determining the value of the ultimate pressure, is the capacity of the pumps employed. For a given value of gas evolution Q (l.mm. Hg/sec.) the ultimate pressure P(mm. Hg) is determined from the equation:

$$P = \frac{Q}{S_e} \qquad (1)$$

where S_e (l./sec.) is the speed at which the system is evacuated and

$$S_e = \frac{S_H}{1+\frac{S_H}{U}} \qquad (2)$$

where S_H is the pumping speed of the pump in l./sec. and U is the conductance of the pipe connecting the pump to the system in l./sec.

It should be noted that the volume of the system does not appear in the equations mentioned. This signifies that the volume of the system has no bearing on the pressure employed in the system. This point is stressed because it has been observed that workers, on their first introduction to the operation of industrial high vacuum plant, often complain about the difficulties experienced in obtaining the low pressures required attributing these difficulties to the size of the system to be pumped. This belief causes errors and unnecessary complications when selecting the plant required for a particular purpose. Details of vacuum apparatus available have been given in various publications dealing with the subject but often the latter are not easily accessible to the workers in industrial laboratories.

The present paper is intended to acquaint the reader with the main characteristics of present-day vacuum pumps, instruments for measuring low pressures and with the design of one or two complete vacuum plants.

Mechanical Pumps

Several types of mechanical vacuum pumps, varying in capacity and designed for a multitude of applications, are produced by long established

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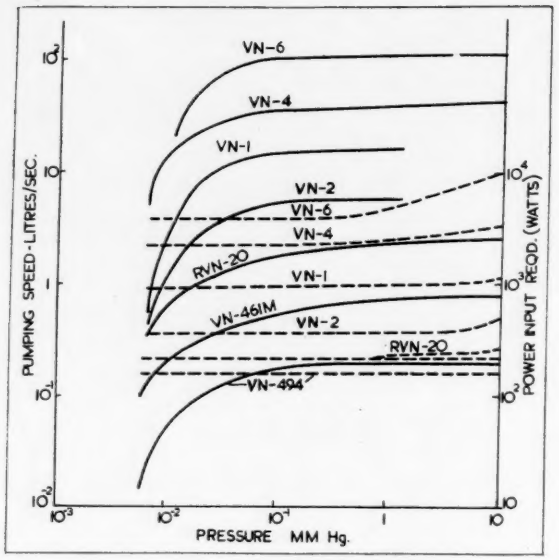


Fig. 1. Relationship between pumping speed, power input and ultimate pressure for various mechanical pumps.

industries. Details of these pumps are given in Table I and Fig. 1. Some are intended to pump down large volumes from atmosphere and others to facilitate the operation of high vacuum diffusion pumps. The construction of these pumps is very similar in all cases, *i.e.* they are all of the rotary oil-sealed type. The pumps have been described at length in a number of publications [1-5].

The VN-494, VN-461M and RVN-20* pumps are intended for use with high vacuum pumps of relatively low output, *i.e.* up to several hundreds of litres per second.

The pumps VN-1 and VN-2[6,7] are also employed mainly as backing pumps for diffusion pumps, but have a fairly high capacity, *i.e.* 500-8,000 l./sec. These pumps cannot be recommended for the pumping down of large vessels from atmospheric pressure as the oil of the pump is entrained by the large volumes of air passing through the pump and after the loss of a certain amount of oil the pump breaks down. For this reason, the factory making the VN-1 and VN-2 pump does not guarantee

* The first two of these are produced by factories of the Ministry of the Electrical Industry, the last by factories of the Ministry of Aircraft Production.

Table I. **Characteristics of Mechanical Pumps**

Designation of Pump		*VN-494*	*VN-461M*	*RVN-20*	*VN-2*	*VN-1*	*VN-4*	*VN-6*
Design Characteristics		*Rotary* — *Rotating vanes*	*Rotary* — *Single vanes*		*Rotary & Piston*			
Pumping speed rated at atmospheric pressure	l./sec.	0.21	0.83	3.3	7	18.3	59	155
Speed of the pump rotor	r.p.m.	360	540	400	525	500	500	360
Ultimate pressure of the pump	mm. Hg	1×10^{-3}	1×10^{-3}	1×10^{-3}	3×10^{-3}	3×10^{-3}	5×10^{-3}	1×10^{-2}
Oil Charge	l.	1.5	2.3	0.5	2.0	3.8	16	55
Method of Cooling			air		air		water	
Cooling water consumption	l./hr.						200–300	700–1,000
Height of the pump	mm.	315	415	445	490	605	1,420	1,975
Length of the pump	mm.	420	610	525	690	910	1,635	1,905
Width of the pump	mm.	235	294	330	560	625	875	960
Weight of the pump	Kg	36	75	110	180	312	1,050	2,050
Electric power required	kW	0.6	0.6	0.8	1.7	2.8	7	20

satisfactory performance of the pumps if operated continuously for long periods at pressures higher than 0.12 mm. Hg. In practice, however, experience has shown that these pumps work for long periods without a breakdown even at pressures as high as 1 mm. Hg. If, in exceptional cases, it is necessary to pump down the whole system from atmosphere, then the intake of the pumps has to be restricted by means of a throttling device in the form of a valve, placed in front of the pump.

The VN-4 and VN-6[8,9] type of pump loses no oil and is suitable for the evacuation of large systems (7-20 m^3). They can operate continuously for long periods at a pressure level of 100 mm. Hg in the region near the pump inlet. The VN-4 pump can also be used as a backing pump to a group of diffusion pumps.

Table II

Type of Pump	*Pumping Time*
VN–6	1 min.
VN–4	3 min.
VN–1	9 min.
VN–2	23 min.
RVN–20	55 min.
VN–461M	3 hr.
VN–494	11.5 hr.

The values shown in Table II give an indication of the time taken by the pumps, mentioned so far, to evacuate a large vessel of given dimensions to a certain value of reduced pressure, if connected directly to the vessel.

These figures demonstrate at the same time that the pumps VN-494, VN-461M and RVN-20 are not suitable for the evacuation of large systems. We shall show that the evacuation of large systems with a pump RVN-20 requires an excessively long time and, in addition, involves a prohibitive loss of oil, as in the case of the VN-1 and VN-2 pumps.

All these mechanical pumps are designed for handling dry, non-condensable and non-reactive gases. The gas passing through the pump should under no circumstances contain any solid particles, as the presence of such particles may damage the pump.

As the use of high vacuum procedures in metallurgical and drying operations continued to spread the need arose for removing not only dry gases, but also mixtures of gas and water vapour. The conventional types of mechanical pumps are not suitable for handling such mixtures as the water vapour entering the pump condenses when its saturation pressure is reached during compression in the pump. The condensate is absorbed by the oil and never leaves the pump. Further, the water present in the oil causes oxidation of the oil and oxidation of the metal parts of the pump which in turn results in excessive wear and tear of the components.

Gas-Ballasting. For the pumping of gas-water vapour mixtures pumps are used fitted with special tanks, where the oil is heat-treated in order to remove the condensed water, or so-called 'gas-ballast' pumps [4, 10, 11, 12] are employed. In gas-ballast pumps the condensation of vapours is prevented by admitting air to the chamber (ballast gas). The air is introduced through an aperture in the end wall of the chamber leading to the atmosphere (see Fig. 2). In Position 1, the aperture *O* is closed by the rotor *P*. At the start of the pumping cycle (Position 2) the aperature *O* is open and atmospheric air enters the chamber. As the rotor continues its movement (Position 3) the aperture *O* is closed and the pressure in the chamber, representing the total of the partial pressures of the pumped vapours and the ballast gas, rises due to the compression taking place until the outlet valve *K* is forced open. To prevent the vapours from condensing during the compression stage prior to their removal to the atmosphere, the ballast gas is introduced in amounts carefully chosen so that the pressure required to open the valve is reached before the partial pressure of the vapours equals saturation pressure. The larger the amount of water vapour in the gas-water vapour mixture, the greater is the amount of ballast gas which should be let into the

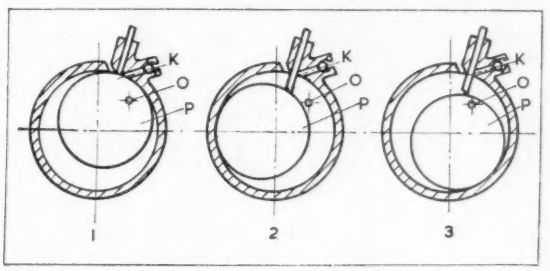


Fig. 2. Diagrammatic indication of the operation of gas ballast pumps.

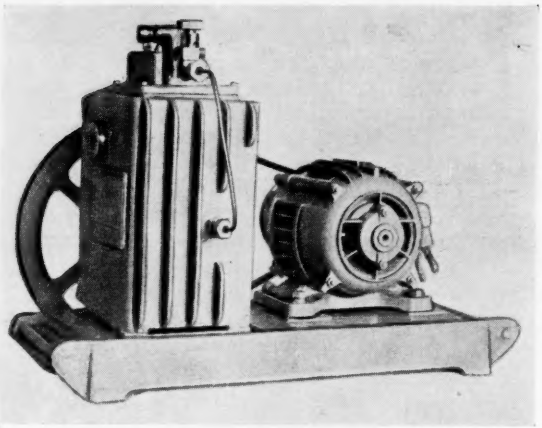
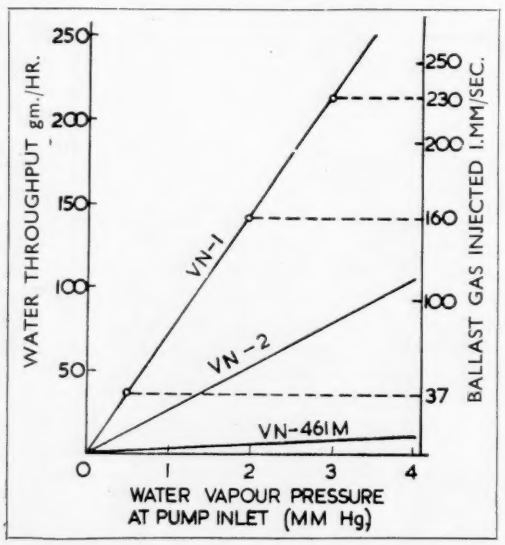


Fig. 3. Pump VN-461M with gas ballast attachment.

pump chamber. The admission of the ballast gas is controlled by a proportioning valve set fitted to the pump, see Fig. 3. The capacities of vacuum pumps suitable for pumping water vapour are indicated by the values shown in graph Fig. 4.

Roots Principle. Recently, as a result of the extensive application of vacuum procedures in metallurgy and drying processes, attempts have been made to raise the efficiency of the mechanical rotary pump in the pressure range of 10^{-1}–10^{-2} mm. Hg, *i.e.* in the range of pressures where the efficiency of the conventional mechanical pump falls off sharply. The American firm Kinney for instance has announced[13] the development of a mechanical pump type KMV-1200 which has a speed of 470 l./sec. in the pressure range of 1-10^{-2} mm. Hg and about 350 l./sec. at 1×10^{-3} mm. Hg. This pump has two stages. The first is built in the form of a Roots air blower and the second operating in series with the first, is of the standard piston-pump type. Pumping from atmospheric pressure down to 15 mm. Hg is carried out by one stage only, *i.e.* the piston stage. Pumping down from 15 mm. Hg is carried out by both stages.

Fig. 4. Water vapour pumping capacity of mechanical pumps fitted with gas ballast attachment.



Diffusion Pumps

For laboratory work, to obtain pressures of 10^{-5}–10^{-7} mm. Hg, mostly mercury diffusion pumps of limited capacity are used, whereas in vacuum processing on an industrial scale the same low pressures are obtained with the help of fractionating oil diffusion pumps which have a speed of several tens of thousands of litres per second. With the latter type of pumps, there is no need to incorporate cold traps in the system in order to obtain these pressures, as the vapour pressure of the pump oils used is very low, about 10^{-9}–10^{-10} mm. Hg at 20°C.

Oil and mercury vapour pumps fall into two categories according to their characteristics.

One category consists of pumps, the maximum pumping rate of which lies in the pressure range of 10^{-6}–10^{-4} mm. Hg requiring a relatively low backing pressure ($\sim$0.1 mm. Hg). The other consists of the so-called booster pumps, the maximum pumping speed of which lies in the region of 10^{-3}–10^{-2} mm. Hg or higher, and which can operate against a backing pressure of up to several millimetres Hg.

Booster Pumps. The vapour-type booster pumps (see Table III) differ little in their construction from high vacuum diffusion pumps.

The jets are somewhat different in design and the pressure of the vapours rising from the boiler is higher than in the normal diffusion pump. The latter is achieved by increasing the power of the heaters and employing a variety of oils which boil readily, as pump fluid.

Pump type BN-3 (see Fig. 5) is used as a backing pump to large capacity diffusion pumps or as the sole means of pumping in vacuum furnaces for the melting or annealing of metals, as well as in drying and distillation plant, etc.

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The factory producing this pump claims that the gas handled by the pump is compressed to not less than 0.3 mm. Hg on the backing side, if a heater of 3.5 kW is used. In actual fact the pressure obtained with a heater of that consumption reaches the value of 0.6 mm. Hg, see Fig. 6. By raising the heating load it is possible to increase the pressure on the backing side to 0.8–1.0 mm. Hg. A change of this kind makes little difference to the speed of the pump, see Fig. 7.

The special feature of the mercury-vapour booster pumps, type DRN–10 and DRN–50, is their high pressure on the backing side. In the case of the DRN–50 pump this reaches a value of 20 mm. Hg. Both pumps can be employed for the same purposes as the BN–3 pump, only the system to be exhausted should be smaller, while the pump DRN–10 could in addition be used in the same way as a conventional high vacuum diffusion pump. There is no point in doing so in the case of the pump DRN–50 as its pumping speed is not high enough (see Fig. 8) at low pressures (~10 l./sec.), in spite of the fact that its size and power requirements are similar to those of a high vacuum diffusion pump with a speed of several hundred litres per second.

High Vacuum Pumps. Modern high vacuum diffusion pumps are made entirely of metal. They are fully reliable to give pressures of 10^{-5}–10^{-7} mm. Hg. The speed of these pumps reaches 20,000 l./sec.

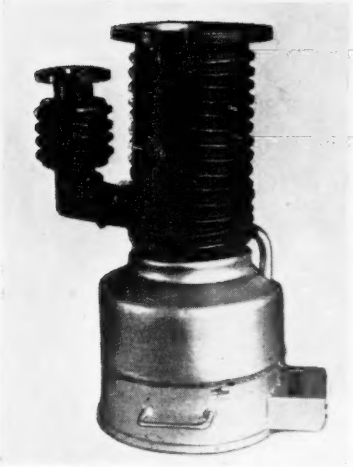

Fig. 5. Booster pump, type BN-3

(see Fig. 9), and there are no principle difficulties in the way of developing and constructing pumps with even greater speeds.

Pumps with a speed of 1,000 to 2,000 l./sec. are very numerous nowadays, let alone pumps of 100–500 l./sec. capacity.

As will be seen from the graph in Fig. 9, the speeds

Table III. **Characteristics of Booster Pumps**

Type		*BN–3**	*DRN–10***	*DRN–50****
Maximum speed	l./sec.	450 at 1×10^{-2}mm. Hg	10 at 1×10^{-2}mm. Hg	30 at 1×10^{-1}mm. Hg
Internal diameter of pump body	mm.	162	35	50
Ultimate pressure	mm. Hg	5×10^{-4}	1×10^{-7}	1×10^{-7}
Maximum backing pressure	mm. Hg	0.3–0.6	1–1.5	20.0
Working fluid		'G' oil****	Mercury R–1	Mercury R–1
Charge of working fluid	ml.	1,500	150	250
Power input	kW	3.5	0.6	2.5
Weight	kg.	33.5	7.5	25
Height	mm.	610	540	875
Area occupied	sq. mm.	413×413	170×170	280×280
Recommended backing pump		VN–1	VN–494	VN–461M

* with liquid nitrogen trap
** produced by a factory of the Ministry of the Electrotechnical Industry
*** produced by factories of the Ministry of the Defence Industry
**** produced by factories of the Ministry of the Fuel Industry

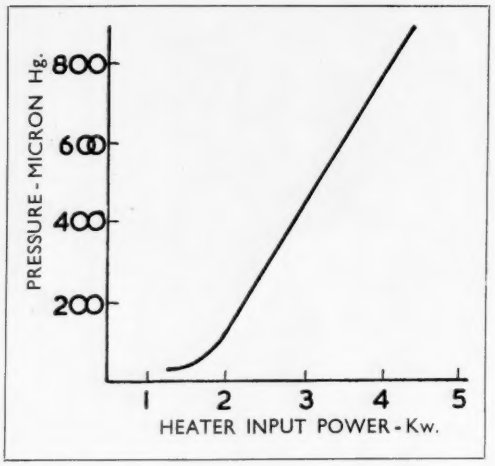

Fig. 6. Characteristics of pump BN-3: Plotting of backing pressure as a function of heater load.

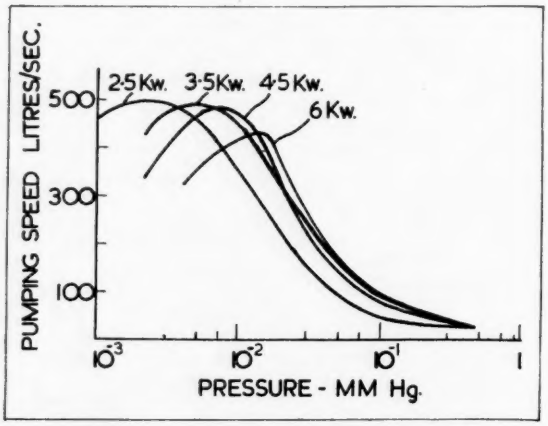

Fig. 7. Characteristics of pump BN-3: Pumping speed for various heater loads in the pressure range 10^{-3}–1.0 mm. Hg.

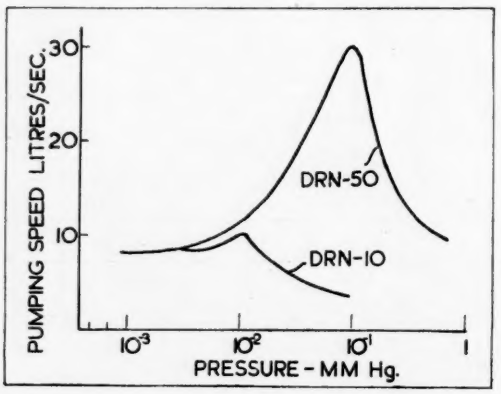

Fig. 8. Characteristics of the booster pumps DRN-10 and DRN-50: Pumping speeds in the pressure range 5×10^{-3}–1.0. mm. Hg.

of these pumps are independent of the pressure over a wide range of pressures.

At pressures below 1×10^{-5} and above $2\text{–}8 \times 10^{-4}$ mm. Hg the speed decreases (see dotted lines). This decrease is due to the fact that at pressures below 10^{-5} mm. Hg, gases evolve from sources inside the pump. At these pressures the walls of the pump liberate gas, and gases constituting the products of the cracking of the working fluid rise with the vapour of the pump fluid. Also there is the phenomenon of back streaming. Therefore, the ' effective ' pumping speed at pressures below 1×10^{-5} mm. Hg depends on the pumping time and the pressure prevailing in the backing line. The better the pump is outgassed and the smaller the leakage, the higher is the 'effective' speed. The horizontal dotted portions of the curves at pressures less than 1×10^{-5} mm. Hg, represent the speed after an extended period of pumping; the curves pointing downwards represent the speed after 5 to 10 hours of pumping.

The pumping speed at pressures above 10^{-4} mm. Hg depends on the design of the pumps. In practically all cases of modern high vacuum diffusion pumps, the horizontal portion of the speed curve of the pumping rate ends in the pressure region of $2\text{–}8 \times 10^{-4}$ mm. Hg.

The characteristics of the more important types of high vacuum oil diffusion pumps are given in Table IV. The first 5 types of pumps, having a capacity of 100, 500, 2,000, 5,000 and 8,000 l./sec. (see Fig. 10), belong to a particular ' family ' of pumps, called the 'single stage' series. The body of all these pumps is made of mild steel, and all are water-cooled. The types N1S and N5S are two-stage pumps, and N2T, N5T and N8T are three-stage pumps. All these pumps are fitted with a device facilitating fractionation of the pump fluid in the liquid phase, which considerably reduces the time required to obtain the ultimate vacuum.

The types TsVL–100, MM40A and N–5*, made of light metal, are also widely used. Pump MM40A is a two-stage pump and has a steel body. Pumps TsVL–100 and N–5 are three-stage pumps. The body of TsVL–100 is of brass, and that of N–5 is of steel. These three pumps are much more complicated in their design than the pumps of the 'single stage'

Table IV. **Characteristics of Metal High Vacuum Pumps**

Characteristics		*N–1S*	*N–5S*	*N–2T*	*N–5T*	*N–8T*	*TsVL–100*
Mean pumping speed for air in pressure range 1×10^{-5} to 2×10^{-4} mm. Hg	l./sec.	100	500	2,000	4,500	8,000	up to 140
Internal diameter	mm. Hg	86	160	260	380	500	86
Ultimate pressure obtainable without liquid nitrogen trap	mm. Hg	3×10^{-6}	3×10^{-6}	3×10^{-6}	3×10^{-6}	3×10^{-6}	3×10^{-6}
Maximum backing pressure	mm. Hg	0.075	0.075	0.1	0.1	0.1	up to 0.2
Charge of D1A oil**	c.c.	50	500	1,000	1,500	4,000	100
Heater load	kW	0.45–0.48	1.0	1.5	2.0	2.8	0.45
Cooling water consumption	l/hr.	50	120	200	350	600	50
Height	mm.	335	478	615	776	1,012	410
Sectional area	mm^2.	175×145	325×260	452×370	596×532	779×650	250×130
Weight	kg	5.5	20	41	79	140	6.3
Recommended backing pump		VN–461M	VN–2 RVN–20	VN–2	VN–1	VN–1	VN–461M

Characteristics		*N–5*	*MM–40A*	*DMN–20*	*N5R*	*N40R*	*N1TR*
Mean pumping speed for air in pressure range 1×10^{-5} to 2×10^{-4} mm. Hg	l./sec.	500	30–40	20	8–10	45–50	1,500
Internal diameter	mm. Hg	162	62	49	35	62	260
Ultimate pressure obtainable without liquid nitrogen trap	mm. Hg	3×10^{-6}	3×10^{-6}	5×10^{-6}	1×10^{-7}*	1×10^{-7}*	3×10^{-7}*
Maximum backing pressure	mm. Hg	0.12	0.05–0.1	0.05	0.6	0.9–1	0.3
Charge of D1A oil**	c.c.	500	40	100	20	50	1,000
Heater load	kW	1.0	0.45	0.2	0.3	0.45	1.0
Cooling water consumption	l/hr.	120	50	air cooled	30	50	300
Height	mm.	600	440	310	200	235	739
Sectional area	mm^2.	350×162	115×120	125×130	100×150	125×168	340×500
Weight	kg	27	6.9	4.2	2.6	3.8	60
Recommended backing pump		VN–2 RVN–20	VN–494	VN–494	VN–494	VN–494	VN–2

* with cold trap
** oil D1A is produced by factories of the Ministry of Fuel Production

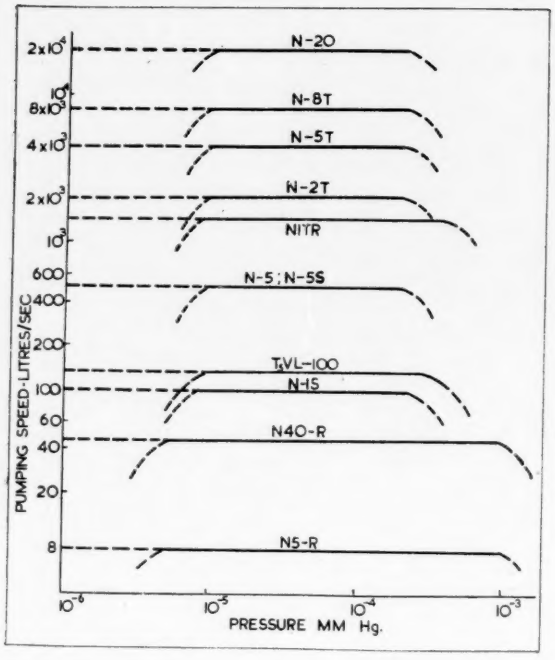

Fig. 9. Characteristics of high vacuum diffusion pumps: Speed *versus* pressure curves for various pumps.

series. They consist of a far larger number of components, which is bound to make the assembly operation and the development work of these pumps much more complex, but they are superior to the 'single stage' series pumps in performance.

In cases, where it is not possible or undesirable to employ water-cooled pumps, the DMN–20* pump is used which is air-cooled (forced air), see Fig. 11. Very large pumping units are fitted with an oil diffusion pump type N–20 of a capacity of 20,000 l./sec. operating with a BN–3 booster pump in series.

For applications where it is undesirable to use oil diffusion pumps, the mercury diffusion pumps N–5R, N40–R and N1TR have been developed by the Ministry of the Radio Industry, see Fig. 12.

A small glass oil diffusion pump, type SDN–1 (see Fig. 13), is produced by the factory of the Ministry of Civil Engineering which works quite satisfactorily. ZS–5 glass is used for the construction. The pump has a speed of about 5 l./sec. Its ultimate vacuum is about $2–5 \times 10^{-6}$ mm. Hg. Maximum pressure on the backing side depends very largely on the characteristics of each individual pump, as well as on the power of the heater. This is understandable considering that glass pumps are built by hand and therefore unavoidably differ in their dimensions from each other. By way of example, we show the variation in maximum pressure on the backing side for two such pumps in Fig. 14.

As working fluid, it is recommended to use 70–100 ml. of D1A oil. The level of the charge should be approximately 0.5 cm. above the heater coils. The optimum heater load lies between 130–150 watt.

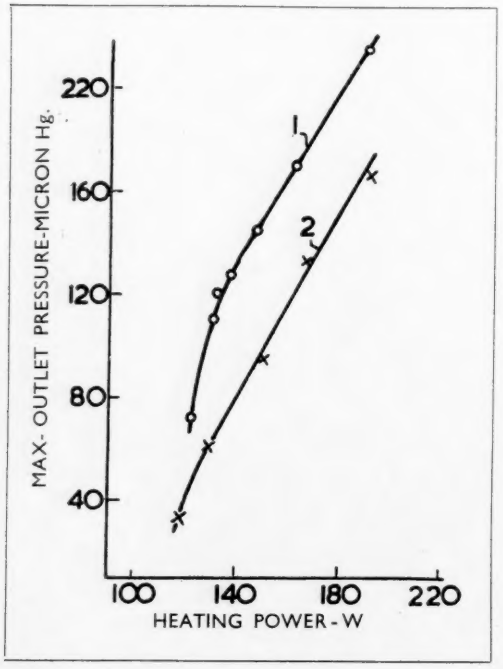

Fig. 14. Backing pressure *versus* heating load characteristics of glass diffusion pump SDN-1.

Pumping Aggregates

Apart from individual components of vacuum apparatus, the following types of complete plant have been developed and are now produced regularly :— complete pumping units, vacuum drying plant, metal sputtering and evaporation equipment [11, 14, 17]. The pumping aggregates represent the basis of any vacuum installation and, therefore, have found widest application. Fig. 15 shows a typical unit of this kind, the VA–05–1 vacuum pumping unit. It consists of a diffusion pump N5S (1), an oil baffle (2), a cold trap

* These pumps are produced by factories of the Ministry of the Defence Industry.

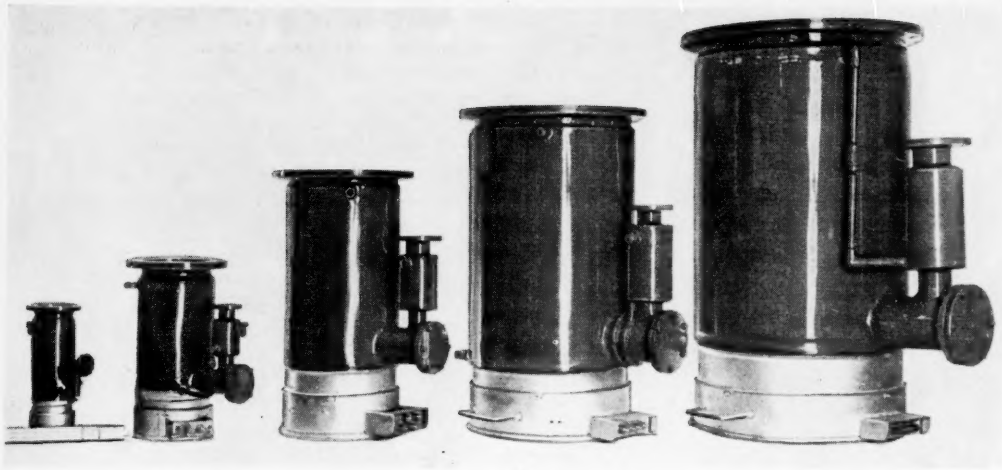

Fig. 10

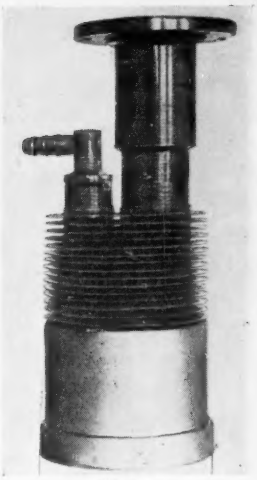

Fig. 11

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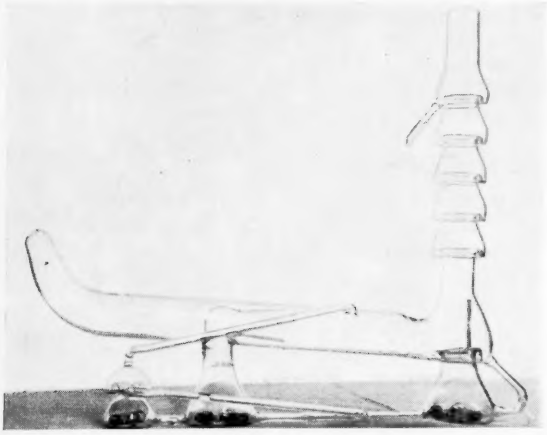


Fig. 13

Fig. 10. High vacuum diffusion pumps.

Fig. 11. Diffusion pump DMN-20.

Fig. 12. Mercury diffusion pumps, types (a) N5R, (b) N4OR, (c) N1TR.

Fig. 13. Glass diffusion pump, type SDN-1.

Fig. 12a

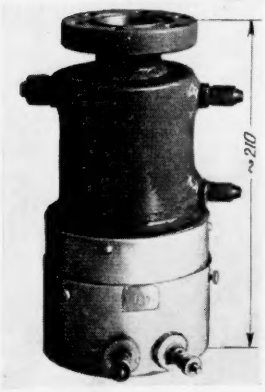


Fig. 12b

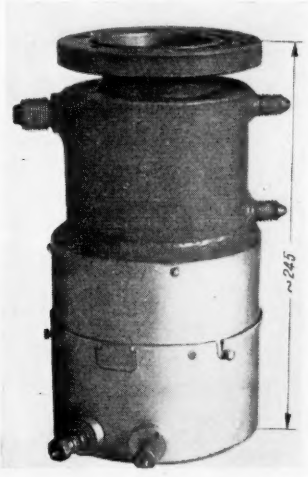
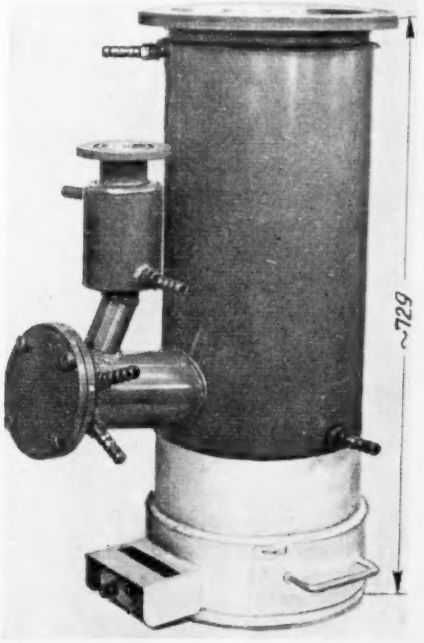


Fig. 12c



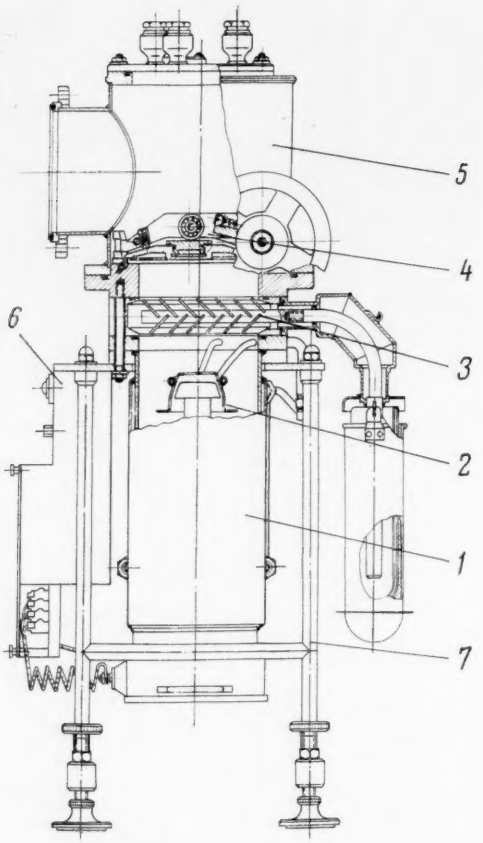


Fig. 15. Diagrammatic view of the pump aggregate VA-05-1.

(3), a vacuum valve (4) fitted to the mouth of the pump (5), and an electrical heater control (6). The unit is connected to a water supply which facilitates the water cooling of the pumps. A supply of as little as 40–50 l./hr. is sufficient. All these parts are mounted on the frame (7). The control panel carries the circuit-breaker and, in addition, a pilot lamp, which is switched off automatically the moment the electric heater becomes overheated, and a bell which gives the alarm when the flow of cooling water is interrupted. The oil baffle (2) is water-cooled and does a great deal to prevent the (back) diffusion of oil vapour from the pump to the system. Its shape is governed by the volume and the speed of the oil vapour constituting the beam emitted by the upper jets in the pump.

Backstreaming of oil vapours into the system is finally prevented by the cold trap (3).

The heat is conducted away from the trap through a copper bar, one end of which reaches into a Dewar flask filled with liquid nitrogen.

Fig. 16 gives a general view of the apparatus VA-01-1 and VA-05-1. Both were developed by the Ministry of Radiotechnical Industry. Table V gives all important data of these pumping units.

Pressure Measurement

Normally in laboratory or industrial operations pressures in the region of 1 mm.–10^{-3} mm. Hg are measured by means of thermoelectric gauges. By special design the range of thermoelectric gauges may be extended to 50–60 mm. Hg[18]. Pressures in the range of 10^{-3}–10^{-7} mm. Hg are measured with ionisation gauges. The standard manometer in the range of pressures from 1 mm.–10^{-5} mm. Hg is the mercury

Table V. **Characteristics of Vacuum Pumping Units**

Type	*Pumping Speed for Air at Pressures* 1×10^{-5}–1×10^{-4} *mm. Hg* l./*sec.*		*Ultimate Pressure* *mm. Hg*		*Charge of Nitrogen in the Trap*	*Each Liquid Nitrogen Charge Lasts*	*Maximum Height*	*Weight*
	With liquid nitrogen trap	*without*	*with liquid nitrogen trap*	*without*	l./*hr.*	*hr.*	*mm.*	*kg*
VA–01–1	30	50	3×10^{-7}	5×10^{-6}	0.12	2.5	800	22
VA–05–1	100	250	3×10^{-7}	5×10^{-6}	0.25	1.0	1,090	75
VA–2–2	500	2,200	3×10^{-7}	5×10^{-6}	1.0	6.5	1,700	240
VA–5–4	1,200	4,500	3×10^{-7}	5×10^{-6}	2	3.5	1,825	360
VA–8–4	2,500		3×10^{-7}	5×10^{-6}	3	2.5	2,080	500

compression manometer, *i.e.* the McLeod gauge.

Several types of vacuum gauges are produced for use in the pressure range of 1 mm.–5×10^{-8} mm. Hg. Of these, the most widely used are the types VT–2, VI–3, VIT–1, VMB–1[12, 19] which have replaced the types UTV–46, UTV–49, VI–1, VK–1. Two varieties are available of these instruments: A portable type and a type suitable for panel mounting.*

The VT–2 instrument, see Fig. 17a, indicates pressures ranging from 1 mm.–10^{-3} mm. Hg. It comprises a gauge head consisting of a thermocouple LT–2 in a glass envelope, or a thermocouple LT–4M in a metal envelope, a source of electric supply and a device for measuring the thermal e.m.f. The gauge has two ranges: A high pressure range from 1 mm.–2×10^{-1} mm. Hg and a low pressure range from 2×10^{-1} to 10^{-3} mm. Hg. Type VI–3 covers the range 10^{-3} to 5×10^{-8} mm. Hg. This instrument consists essentially of an ionisation gauge head type LM–2 and an electrical measuring and control circuit. The instrument incorporates a ferro-resonance voltage stabiliser, giving it greater operating stability. In addition, it has an electro-magnetic emission current stabiliser which considerably eases the use of the apparatus. The indication of the instrument is linear.

When using the VI–3 gauge the following points should be borne in mind :—

(1) If the ionisation gauge head (LM–2) is operated for long periods at pressures of the order of 10^{-3} mm. Hg, the cathode will rapidly deteriorate, but if the tube is operated at pressures not exceeding 10^{-5} mm. Hg it will last for hundreds of hours. Admission of atmospheric air will also destroy the cathode.

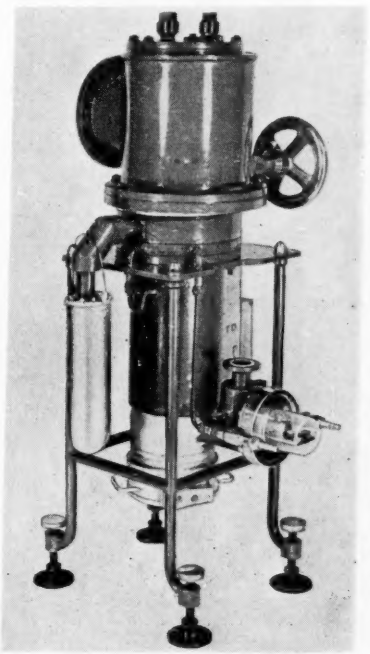
(2) Since in the majority of cases the composition of the gases measured is not known, care should be taken in interpreting the gauge indication as the instrument is calibrated against dry air.

The VIT–1 gauge, see Fig. 17b, represents a combined manometer covering the full range from 1 mm.–5×10^{-8} mm. Hg. The detector elements are the ionisation gauge head LM–2 and the thermocouple LT–4M, or LT–2.

The calibration curves given for the LT–2 and LT–4M elements apply to dry air. The deviation from the calibration curves does not exceed $\pm10\%$ in the case of LT–2 and $\pm20\%$ in the case of LT–4M so long as the pressure of dry air is measured, but if the pressure of other (pure) gases is to be determined in the range from 10^{-1} to 10^{-3} mm. Hg and the

* The panel type is distinguished from the portable type by the letter ' P ' (VT–2P, BI–3P, etc.)

Fig. 16. Vacuum pumping unit, type (a) (above) VA-01-1 and (b) VA-05-1 (below).



a b c

Fig. 17. Low pressure reading instruments: (a) VT-2 with thermocouple detector element LT-4M, (b) VIT-1 with thermocouple detector elements LT-2 and LM-2, (c) VMB-1 with thermocouple detector element MM-5.

●

Table VI. **Physico-Mechanical Properties of Special Rubbers for Vacuum Use**

Properties		*Rubber* 7889	*Rubber* 9024
Tensile strength	kg/sq.cm.	170	100
Elastic strength	%	550	350
Permanent stretch (max.)	%	20	14
Jones hardness	kg/sq.cm.	4.5–7.5	7–11
Swelling after 24 hr. immersion in vaseline oil at 70°C.	%	90	6

●

Table VII. **Dimensions of Rubber Sheets**
(Rubber 7889 and 9042)

Sheet thickness mm.	*Length in mm.* *A*	*B*	*Width in mm.* *A*	*B*
2	250	1,500	250	750
3	250	1,500	250	750
3.5	500	1,500	500	750
5	500	1,500	500	750

thermocouple LT–2 is employed, a coefficient has to be used to evaluate the readings correctly. Values for this coefficient are given in Khabkin's article[20].

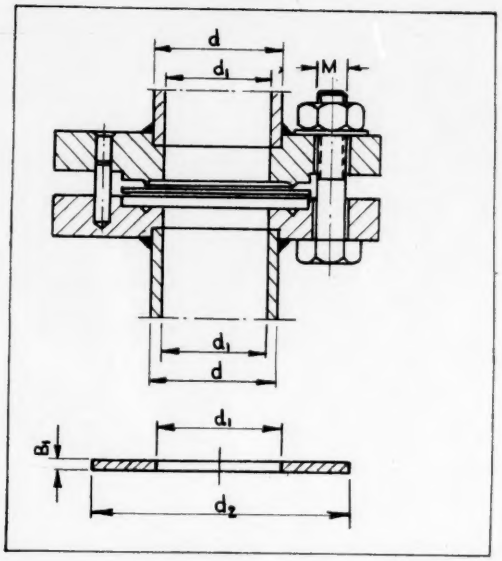
The VMB type of gauge, see Fig. 17c, measures air pressures from 8×10^{-4} to 2×10^{-6} mm. Hg, and, in addition, is designed to 'control' pressures in the range of 5×10^{-4} to 5×10^{-6} mm. Hg. It comprises a Penning-type detector element MM–5, an electric supply for the detector element and a relay circuit effecting the pressure control. The accuracy of this instrument is $\pm35\%$. By means of the vacuum control system of the apparatus, it is possible to select any given values of pressure in a certain range, for carrying out a process. The relay of the vacuum control system facilitates switching external circuits operating at 220–500 V. Penning-type manometers incorporating the MM–5 gauge head have no hot cathodes and thus are not damaged when exposed to atmospheric air. Thus these instruments are highly suitable for application in industrial operations.

The description of Knudsen-type manometers, alphatrons, mechanical pressure gauges and gauges for ultra high vacuum [3–5, 21–24] is outside the scope of this article.

Joints and Seals

The majority of vacuum apparatus is assembled from a number of components and thus they possess many permanent and demountable joints. The joints are sealed with the help of gaskets made from metal or a special sort of rubber or plastics which have a low gas evolution. The rubber most widely used for the purpose are the types 7889 and 9024. The physical and mechanical properties of these materials are shown in Table VI.

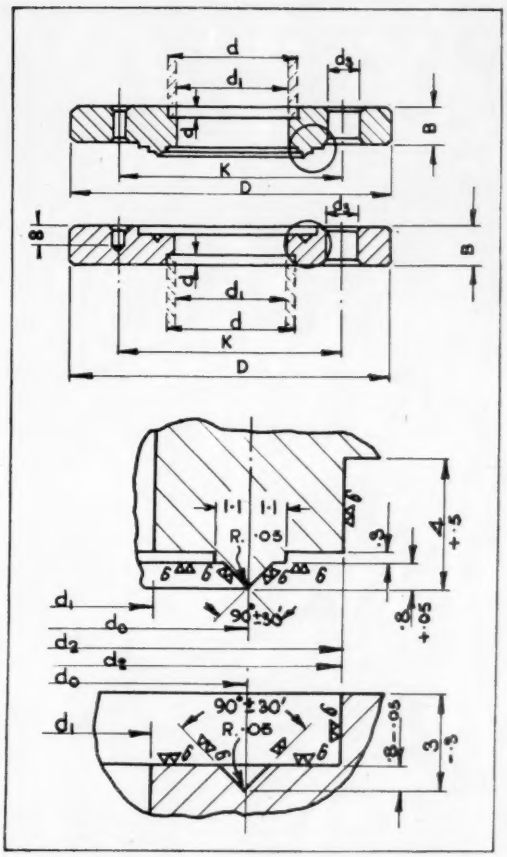
Rubber 9024 is used where permanent joints are



sealed with grease, as it has a higher resistance to oil than rubber 7889. Rubber 9024 is produced in the form of sheet (TU MKhP No. U251–54). Rubber 7889 is also available in the form of sheet (TU MKhP No. U251–54), and in addition, in the form of cord with a rectangular or a round section (TU MKhP No. U213–54) and in the form of tubing (TU MKhP No. 1472–54). The latter is mainly used for ' rough-pumping ' lines.

Cords with a rectangular or a round cross section are produced in thicknesses of 3 to 30 mm. The internal diameter of the tubes available is 3, 6, 9, 12, 15 or 30 mm. The thickness of the tube wall is the same as the internal diameter. The most popular sizes are 9, 12, and 15 mm. Tubing of this size is employed for the backing lines connecting the diffusion and the mechanical pumps type VN–494, VN–461M and RVN–20. Sheets are produced in two different ways. Sheets of type A are moulded and joints are effected by vulcanisation under pressure. The surface of these sheets is as smooth as that of the mould employed in the process. Sheets of type B are rolled and joints are effected by vulcanisation while the sheet is still on the roller. Here the surface of sheets shows a similar texture to that of the roller surface. The sizes of the sheets which are most frequently used in practice are given in Table VII.

Gaskets made from B–type sheet are used with success in metal apparatus. Gaskets made from A–type sheet are used to seal glass panels to metal



Figs. 18a and b. Vacuum-tight flanged joints with metal gasket.

Fig. 19. Vacuum joints with rubber ring as sealing element, (a) permanent seal and (b) demountable joint.

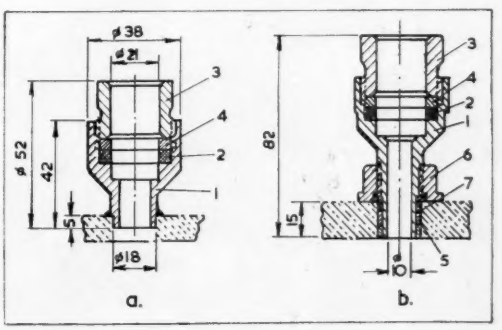
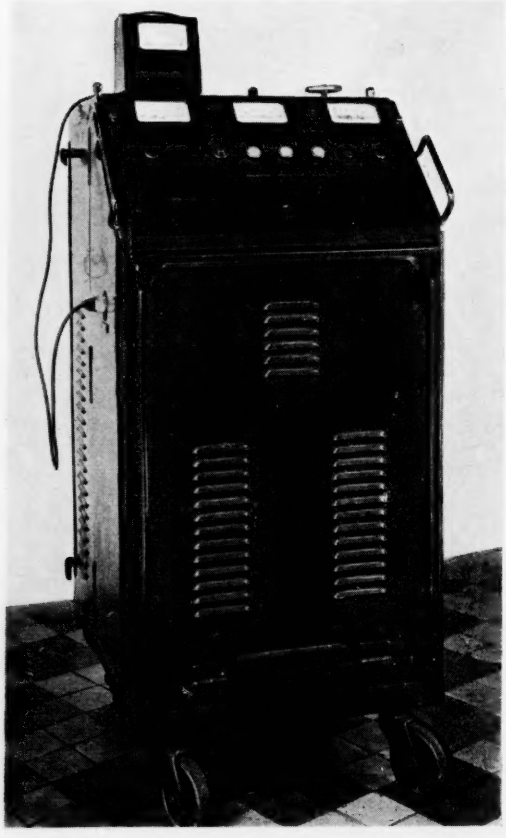


Table VIII. **Dimensions of Flange Joints with Metal Gaskets (mm.)**

d_1	d	d_2	d_0	D	k	a	b	d_3	b_1	M	i
10	14	20	15±0.1	58	40	3	10	9	0.4	8	4
15	19	25	20±0.1	65	45	3	10	9	0.4	8	4
20	24	30	25±0.1	73	55	5	12	11	0.4	10	4
25	30	25	30±0.1	85	62	5	12	11	0.4	10	4
40	45	50	45±0.1	110	80	5	14	13	0.4	12	8
70	76	80	75±0.15	145	115	5	15	13	0.4	12	8
150	156	170	160±0.2	230	195	8	22	13	0.5	12	12
200	206	220	210±0.2	280	250	8	24	13	0.5	12	16
306	306	320	310±0.25	380	350	10	28	13	0.5	12	24

NOTE: Material for flanges and bolts, 20 Steel (GOST 1050-52) or stainless 1Kh18N9 or 1Kh18-N9T (GOST 5632–51) steel: For nuts and pins, 45 Steel (GOST 1050–52): For copper M1 (GOST 859–41) soft cold-rolled sheet (GOST 495–50) or aluminium A1 (GOST 3549-47) soft sheet (GOST 1946-50).



such as observation windows, for instance, and where, due to the simple construction of the apparatus, only moderate pressures can be applied to effect proper sealing. In cases where areas of a large perimeter have to be sealed, it is cheaper to use cord. The cord is stuck to the surface with gum, but if the joint is dismantled frequently, it is not necessary to employ an adhesive. Seals made from cord of rubber 7889 are quite reliable.

Seals of this kind remain intact for several years, and do not need any mechanical support (*e.g.* the provision of bolts). Various methods to effect a seal with rubber gaskets in pipe joints for pipes of 10 to 500 mm. diameter are described in a book published a short time ago[12].

In cases where it is not possible to use rubber gaskets (because the apparatus is heated, for instance), metal gaskets are employed. The design of a joint with metal gaskets is shown in Fig. 18, and its basic dimensions in Table VIII. The gaskets are ring-shaped stampings, made from cold-rolled copper sheet M1 or aluminium sheet A1. The copper gaskets should be annealed and the scale should be removed before use. Vacuum seals with metal gaskets can withstand heat up to 400°C.

An excellent material for gaskets from the point of view of vacuum engineering, is fluorine plastic[25]. Fluorine plastic is chemically stable over a wide range of temperatures, and is not affected by heat up to 250°C. Its gas evolution is far lower than that of rubber. In many cases, especially when working with

Fig. 20. Leak detector, type PTI-4A.

reactive gases, its use is indispensable.

Where glass components are to be fitted to vacuum apparatus constructed from metal, special joints have been developed (see Fig. 19) which employ a rubber ring as the sealing element. A permanent joint of this kind is shown in Fig. 19a, depicting the method of connecting a gauge to the system. To obtain air-tightness the rubber ring (2) in joint (1) is compressed by the nut (3). The washer (4) protects the rubber ring against tearing due to the movement of the nut. Fig. 19b shows a demountable joint built in accordance with the same principle, normally used when the connection can neither be soldered nor welded to the apparatus. The mushroom-shaped casing fits into a hole in the wall of the apparatus and vacuum-tight sealing is effected by means of the rubber ring (5) compressed by the nut (6). The washer (7) protects the ring from tearing.

Methods of Leak Detection

The most sensitive method of detecting leaks in vacuum apparatus is the mass-spectrometer method. The method is based on the detection of a probe gas entering the apparatus at the point of leakage in an instrument working on the principle of the mass-spectrometer[12, 26]. The mass-spectrometer leak detector PTI–4A (Fig. 20) is intended to be used with helium as the probe gas. Helium is particularly suitable for the purpose because of its low molecular weight, its low concentration in air, and because no explosion hazards are involved in handling it.

The sensitivity of leak detector PTI–4A is so high that it records even the amount of helium present in atmospheric air, the hand moving across some 10–20 divisions of the scale of the instrument.

In practice, using the leak detector PTI–4A correctly, it is possible to trace leaks of the order of 10^{-4} μl./sec. Assuming the vessel has a volume of 10 litres and is evacuated to 0.001 mm. Hg this order of leakage, if not attended to, would cause a pressure increase in the vessel to 1 mm. Hg in the course of approximately 3 years.

However, the detection of such small leaks requires special methods even where the detector PTI–4A is employed. These methods are described in detail in a book dealing with the fundamentals of vacuum practice[12]. The mass spectrometer leak detector must be handled by a skilled operator. Otherwise even large leaks will remain undetected.

Small leaks can also be traced with the 'halogen' leak detector which has the same degree of sensitivity as the mass-spectrometer leak detector. Here, a halogen is used as the probe gas. The halogen containing air or vapour passes over the surface of platinum maintained red hot. As a result the emission of ions from the platinum rises sharply and this effect is utilised to indicate the presence and size of a leak.

The halogen leak detector is shaped like a pistol. The air and halogen containing vapours are drawn through the 'barrel' of the pistol. A small valve fitted to the pistol controls the flow of the latter.

Other laboratory methods of leak detection include the application of high pressure or high frequency (Tesla coil), or the use (in a special manner) of thermo-electric and ionisation gauges, in particular the so-called palladium leak detector[26, 27], and others, but all these methods are less sensitive than the two instruments described above. Also their range of application is far more restricted.

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Moscow, Russia. December, 1955.

References

1 IVANOV, A. A., *Electric Vacuum Technology*, (Gosenergoizdat, USSR, 1944).
2 KOROLEV, B. I., *Fundamentals of Vacuum Technique*, (Gosenergoizdat USSR, 1950).
3 GUTHRIE, A. and WAKERLING, R. K., *Vacuum Equipment and Techniques*, (McGraw-Hill Publishing Co., Atomic Energy Series, 1951).
4 JAECKEL, R., *Kleinste Drucke, Ihre Messung und Erzeugung*, (Springer-Verlag, Berlin, 1950).
5 DUSHMAN, S., *Scientific Foundations of Vacuum Technique*, (John Wiley & Sons, Inc., New York, 1950).
6 *Instruction of Ministry of Industry, USSR*, (1953). 'Vacuum Pump Type VN-1'.
7 *Instruction of Ministry of Industry, USSR*, (1954). 'Vacuum Pump Type VN-2'.
8 *Instruction of Ministry of Industry, USSR*, (1954). 'Vacuum Pump Type VN-4'.
9 *Instruction of Ministry of Industry, USSR*, (1954). 'Vacuum Pump Type VN-6'.
10 KASSNER, D. A. and LASKY, C., *Rev. Sci. Instrum.*, **24**, (1953), 832.
11 HOLLAND-MERTEN, E. L., *Handbuch der Vakuumtechnik*, (Veb Wilhelm Knapp Verlag, Halle, 1953).
12 LANIS, V. A. and LEVINA, L. E., *Practical Fundamentals of Vacuum Investigations*, (Gosenergoizdat, USSR, 1955).
13 *Nucleonics*, **12**, (1954), No. 6, 87 and No. 12, 57.
14 OSHCHEPKOV, P. K., REIKHRUDEL, E. M. and STASIUK, E. M., *Zavodskaya Laboratoriya*, **19**, (1953), 87.
15 FRIMER, A. I. and NEFELOV, A. K., *Izsestiya Akad. Nauk, USSR, (Phys. Ser.)*, **15**, (1951), 336.
16 HOLLAND, L., *J. Sci. Instrum. Suppl. I (Vacuum Physics)*, (1951), 59.
17 MALCOLM, E., *ibid*, 63.
18 MIRTOV, B. A., *Zhur. Tekh. Fiz.*, **25**, (1955), 726.
19 TURCHININ, A. M., *Electrical Measurement of Non-Electric Valves*, (Gosenergoizdat, USSR, 1954).
20 KHAVKIN, L. P., *Zhur. Tekh. Fiz.*, **25**, (1955), 726.
21 STECKELMACHER, W., *J. Sci. Instrum. Suppl. I.*, (1951), 10.
22 *Rev. Sci. Instrum.*, **21**, (1950), 580, 941.
23 ALPERT, D., *J. Appl. Phys.*, **24**, (1953), 860.
24 KELLY, F. M., *Rev. Sci. Instrum.*, **21**, (1950), 673.
25 GARBARA, M. I., Editor, *Fluorine Plastics*, (Goskhimizdat, USSR, 1954).
26 LEVINA, L. E., *Uspekhi Fizicheskikh, Nauk*, **22**, (1955), 101. 'Present-Day Methods of Leak Detection'.
27 BLEARS, J., and LECK, J. H., *J. Sci. Instrum. Suppl. I.* (1951), 20.

LETTERS TO THE EDITOR

A Method for Protecting the Surfaces of Some Materials in Freeze Drying

Sommaire

UN MANQUE D'HOMOGENEITE de la texture près des surfaçes et une adhésion solide sur les plateaux de quelques matériaux apparaissent à la lyophilisation des purées de fruit. Ce phenomène est évidemment du à une fonte partielle du produit. Ces difficultés ont été évitées en formant des couches fines de glace pure au dessus et au dessous du produit à lyophiliser.

In the preparation of freeze-dried materials containing large quantities of sugar it is recognised that there are difficulties in obtaining a hómogeneously dried product. Non-uniformity in the dried material was observed when freeze drying apricot and peach purées in trays ($10\frac{1}{4}\times6\frac{3}{8}\times1\frac{1}{8}$ inch.) fitting a medium size freeze drying cabinet holding 32 trays each heated electrically. The purées were frozen before loading into the drier and about 50 minutes were required to reduce the pressure to 0.1 mm. Hg. Under these conditions thawing of top surfaces often occurred, causing frothing, and the formation of gummy films of the processed material which restricted drying and gave a dried product which was not uniform in texture. In addition, some gummy material was found adhering to the bottom of the trays, possibly due to partial thawing towards the end of the sublimation stage. The latter could not be removed completely after processing and, since it was probably different in composition from the bulk of the end product, sampling of the material for chemical studies was unsatisfactory.

A simple method of overcoming these difficulties is to form ice layers above and below the material in the freezing stage. The trays are placed on a level shelf in a freezing chamber at −20°C., and a charge of distilled water and purée in separate trays are first brought down to a temperature of 1°C. Then enough water is poured into trays to form an ice layer 1/10 to 1/8 inch. thick. On this a layer of fruit purée, from 1/4 to 3/8 inch. thick, is poured and levelled as far as possible by shaking and tapping, and then allowed to freeze. A final ice layer 1/10 to 1/8 inch thick is formed on top of the purée but the thickness of this may have to be larger in places to smooth out irregularities in the top surface of the purée.

Although this method increases the amount of ice to be sublimed by about 50 to 100%, other techniques for obtaining thinner ice layers have not been tried. It is presumed that the need for the ice layer on top would not arise in freeze driers where independent cooling and heating of the shelves permits pre-freezing *in situ*, but the bottom layer might still be useful. In a freeze drier lacking these facilities the method gives dried materials of uniform texture and appearance which can be easily removed from the trays.

The work described in this paper was carried out as part of the programme of the Division of Food Preservation and Transport of the Commonwealth Scientific and Industrial Research Organization, Australia.

J. D. MELLOR.

Division of Food Preservation,
C.S.I.R.O.,
Homebush, N.S.W., Australia.

20th February, 1956.

Rubber in Vacuum Systems

Sommaire

ON A OBSERVE UNE décoloration du cuivre employé dans un système à vide, en presence de quelques types de coutchouc, bien qui le coutchouc naturel ou au silicone semble être inoffensif. Ce phenomène n'est observé que sur une pièce de métal: les films de cuivre évaporé semblent être insensibles.

The contact-staining of metals by some rubbers is well-known. We have noticed, amongst the usual distributions in bell jars after vacuum processes, a preferential discoloration of previously clean copper, remote from any rubber in the system, usually attributed to cracked oil. This is observed to occur at room temperatures. The colour and thickness of the deposit depends upon time and pressure. At 5×10^{-2} mm. Hg the deposit is very thin, reddish purple and takes an hour or so to develop, whereas a few minutes at 5×10^{-5} mm. Hg produces a green-blue scale. We found this was due to rubber present in the system but not in contact with the copper.

It is possible that other metals are contaminated under vacuum conditions by rubber. We tested platinum, steel, tungsten, molybdenum, tantalum, brass and silver, giving exposures of an hour at pressures

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of the order of 10^{-5} mm. Hg, without obvious change of surface colour.

Chemical analysis of the fresh discoloration upon copper indicated a sulphide, although after exposure to air there was a marked darkening of the deposit and electron diffraction tests suggested a surface layer of copper oxide. The obvious sources of sulphur were the rubber gaskets used in the system although there were no outgassing difficulties indicated by the gauges. The colour effects were obtained equally rapidly with a mercury diffusion pump and with oil pumps, which indicated that oils were not responsible.

A 6-inch. diameter bell jar was sealed to our system with Apiezon 'Q' and clean copper foil was exposed under vacuum. No discoloration occurred in one hour and we were able to do comparative checks on different rubbers placed in the system.

A small piece ($1\times\frac{1}{8}\times\frac{1}{8}$ inch.) of L-gasket rubber placed in the bell jar (sealed with Apiezon Q) was evacuated to about 5×10^{-5} mm. Hg in 15 minutes. In a total time of 20 minutes the copper foil was blue-black. Various types of rubber were tested, and under these conditions we found natural rubber and silicone rubber apparently satisfactory, in that no coloration occurred.

We then found that a material could pass the above test, but if made into a gasket for sealing purposes would still produce the effects, *i.e.* when under sufficient compression. It is important in most process plants that objects made of cleaned copper should not spoil if left under vacuum conditions and the suitability of a gasket rubber can be checked in a few minutes in a system using a small piece of copper foil as the detector.

The effects on copper referred to above are not observed if the copper is present in the form of an evaporated film, only if it is present as bulk material. The reason for this immunity of evaporated films is not known. Also it has been noticed that, if in actual contact with rubber, such films are attacked.

Thanks are due to Mr. G. W. Milner of the Chemistry Division for assistance with the analysis. Messrs. Midland Silicones, I.C.I. and Dunlop kindly provided rubber samples.

J. G. S. Biram & T. A. J. Jacques.

Atomic Energy Research Establishment,
Harwell, nr. Didcot,
Berks., England.

16th July, 1956.

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BOOK REVIEWS

High Vacuum Technique. (3rd Revised Edition.) By J. Yarwood (Chapman & Hall Ltd., London, 1956.) Pp. 208. Price £1 10s. 0d.

During the last decade we have seen an enormous increase in the applications of high vacuum technique to an ever widening field. So extensive is this application today that there is scarcely a branch of applied science that may be studied without a basic knowledge of the art. Indeed many science syllabuses call for a specific treatment of this important facet of modern applied Physics. For this reason the present little book by J. Yarwood is most valuable, forming as it does, both an introduction to the subject and also a book of reference.

It is a pity, however, that in this third and latest edition the opportunity was not taken to bring the work up to date by the omission of apparatus and techniques which are now of little more than historic interest. As it is, the newcomer to the subject is liable to be left with a false impression of current techniques. For instance, the author stresses the use of glass in vacuum systems, but in modern practice metal is used wherever possible because of its ease of fabrication and ruggedness. Modern fractionating pumps are not mentioned and the subject of leak detection which in recent years has advanced so much is only briefly referred to. Many of the diagrams are also out of date and in some cases inaccurate. Fig. 33 is described as a 'typical pumping system'. It is in fact the same diagram as appeared in the first edition and was at that time a typical system. The diagram of Fig. 70 is confusing, particularly as the voltage stabiliser appears to be connected directly across the high tension supply.

The 'lusec' which is now the accepted unit of throughput is scarcely mentioned in the book. There is a footnote on page 105 but this serves only to con-

fuse the reader. Similarly confusion occurs at the foot of page 59 in connection with the use of rubber gaskets. Here the careless use of the term total pressure has involved the author in trouble particularly as he proceeds, quite rightly, to define pressure as force per unit area.

The section dealing with the measurement of high vacuum contains a mass of miscellaneous information on gauges, which, whilst covering the theory of operation satisfactorily, says little to guide a newcomer in the selection of equipment. The author seems unaware that vast improvements in the regulators for use with ionisation gauges (Steckelmacher, 1950) have made obsolete the circuit he describes and it seems a pity that the newer circuit could not be substituted for the old.

No mention is made of the precautions to be taken in using the gauges (outgassing, etc.) or to compare their advantages and disadvantages. The reader may also be surprised at the cost of regulator units which are nowadays far greater than that of the gauge head itself.

Pump speed measurements are fully treated and followed by a chapter on degassing and gettering. This deals with the requirements for the valve industry but omits all mention of the difficulties of gas removal in vacuum melting and other industrial applications. The applications themselves are barely outlined and give no idea of the extent, to which vacuum evaporation and sputtering are employed. The technique of 'blooming' glass and the preparation of dichroic filters are not mentioned; the only references are to Strong (1936) and O'Bryan (1934).

The final 40 pages consist of a list of materials and properties which would be more useful if it included details of suppliers.

In spite of the above criticism however, the writers have no hesitation in recommending this work as a useful addition to any science library.

J. EWINS, A. WALSH.

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. . . *of vacuum interest*

Lectures

THE ENFIELD TECHNICAL COLLEGE, QUEENSWAY, ENFIELD MIDDX., has announced a special lecture series on HIGH VACUUM TECHNIQUE to be held on Tuesday evenings commencing on the 22nd January, 1957 at 7 p.m. and consisting of 12 lectures. The lecturer is A. Williams, B.Sc.

Subject of Lectures:

Kinetic Theory of Gases.
The Measurement of Low Pressure.
Production of High Vacua.
The Flow of Gas Through Tubes.
Vacuum Plumbing.
Gas-Solid Phenomena.
Industrial Applications.

The fee for the course is £1

FURTHER LECTURES are announced as follows:

Tuesday, 16th October, 1956 at 5.30 p.m.
High Vacua.
Lecturer: *M. E. Haine.*
Held at: 47, Belgrave Square, London, S.W.1.
Arranged by: The Institute of Physics, Electronics Group.

Thursday, 22nd November, 1956 at 7.0 p.m.
Titanium.
Lecturer: *N. P. Inglis.*
Held at: Constantine Tech. College, Middlesbrough.
Arranged by: Society of Chemical Industry, Newcastle Section and Royal Institute of Chemistry.

Wednesday, 12th December 1956, at 6.30 p.m.
The Preparation and Testing of Diffraction Gratings.
Lecturer: *L. A. Sayce.*
Held at: 47, Belgrave Square, London, S.W.1.
Arranged by: Institute of Physics.

Tuesday, 26th March, 1957 at 6.15 p.m.
The Supply and Distribution of Liquid Helium.
Lecturer: *E. Mendoza.*
Held at: New Hall, Royal Horticultural Society, Westminster, London, S.W.1.
Arranged by: The Physical Society.

Thursday, 2nd May, 1957 at 2.30 p.m.
Protective Coatings for Thin Films.
Lecturer: *L. Holland et al.*
Held at: Imperial College, South Kensington, London, S.W.7.
Arranged by: The Physical Society, Optical Group.

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Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

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Filing of Abstracts

All pages containing the text of abstracts are printed on one side only and are detachable ready for filing in special folders. Each main group of subjects (I, II, III, IV) starts on a separate page in each issue and all abstracts in any one group are numbered consecutively throughout all issues of a volume. To distinguish the current numbers of abstracts belonging to one of the four groups from those allocated to other groups, the group number is shown as a suffix to the current number of the abstract, *e.g.*, 20/I as distinct from 20/II, etc.

Translations

Translations of some of the foreign publications quoted can be supplied.

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10 — VACUUM ENGINEERING (GENERAL) — 10

Design of a Large Vacuum Vessel 136/I

See Abstract No. : 162/I

A Graphical Approach to Vacuum Engineering 137/I

United States. A new technique is presented for the graphical solution of the steady state and transient flow of gases in a vacuum system throughout all the pressure ranges normally encountered. Methods are established for the analysis of various components as well as complete systems. Evaluation of such items as pumping-down time, time response, leakage and gas flow conversion are also discussed. The paper is based on a lecture contributed by the authors to the Second Symposium held by the Committee on Vacuum Technique in 1955 but also contains additional information which has been compiled during studies of the subject in subsequent months. Copies of Vacuum Engineering Graphs, marked FN 756A and FN 756B are available from the General Electric Company.

Article by D. J. Santeler & J. F. Norton *Vacuum* 4, April 1954 176-194

O *Sommaire* : Présentation d'une technique pour la solution graphique des gaz à l'état stable et instable dans un système à vide, ceci dans toutes les gammes de pression.

Ionic Pump 138/I

See Abstract No. : 92/II

The Physical Processes in Diffusion and Ejector Pumps 139/I

Germany. The working principle of the diffusion and the ejector pump is the same: Gas at low pressure is conveyed by a vapour stream to an area of high pressure. Their characteristics are defined by three qualities: Pumping speed, ultimate vacuum obtainable and fore vacuum requirements. The construction of the two types of pump is very much alike. The essential parts are a boiler, a Laval-type jet, a mixing space and means for condensing and returning the fuel to the boiler. The gas to be pumped is entrained by the (fuel) vapour stream in the mixing space and liberated in the area where the fuel is condensed to be taken up by the fore vacuum pump. But their performance characteristics vary appreciably. The vapour pressure of the fuel in the boiler of the ejector pump is very much higher than that of the fuel in the diffusion pump. As a result the ejector pump works in a higher pressure region on the inlet and on the outlet (fore vacuum) side of the pump than the diffusion pump. Plots of pump speed *versus* (vacuum) pressure in the case of the ejector pump show, with increasing vacuum pressure, a rapid rise up to a maximum followed immediately by a sharp reduction, whereas the speed curve of a diffusion pump shows a typical plateau signifying constant maximum speed over a considerable range of pressures before, at the high pressure end of the range, the speed is reduced. However, by varying the heater load the speed curves of both types of pump can be made to look very similar and thus their normal performance characteristics merely represent the extreme limits of one and the same pump. In order to gain information on the pumping mechanism in both pumps the flow conditions prevailing in the mixing area have been studied. Mercury was used as fuel. To facilitate visual observation the mixing chamber was made of glass and an electrical discharge created in the mercury vapour issuing from the nozzle. Details of the experimental
O set-up are given. It was found that the speed of the vapour leaving the jet is supersonic at all pressures. The shape of the vapour stream is conical, the width of the cone base depending on the pressure prevailing in the mixing area. The static pressure and the Pitot pressure on the axis of the vapour cone were measured by means of a special probe, details of which are given. A steady pressure drop was measured up to the point where the cone reached its maximum width, *i.e.* the width of the mixing area in the case of suitable pressure conditions. Measurements beyond that point showed a steady rise of the pressure. The actual mechanism of entrainment was studied using oil as the working fluid, pumping a mixture of oxygen and nitrogen. The latter was passed through an electric arc immediately prior to entering the mixing chamber so that the after-glow obtained facilitated visual observation. In proper vapour jet pumps operating at comparatively high pressures mixing is the result of turbulence. In the pressure range investigated here the Reynolds number is too small and mixing is solely due to diffusion of the pumped gases. The experiments showed that in the case of the diffusion pump, operating at very low pressures, diffusion is complete, but in the ejector pump, operating in a higher pressure range, the gas penetrates the vapour stream only to a limited depth. The experiments permit the following conclusions: In the case of the diffusion pump the gas pressure on the inlet side, *i.e.* in the mixing chamber is negligibly small compared with the vapour pressure of the fluid. Thus the dispersion of the fluid leaving the nozzle is independent of that pressure. The motion of the gas particles reaching the mixing area is typical of gas diffusing through an orifice into a vacuum, *i.e.* diffusion is complete and the amount of gas in terms of volume per unit time entrained by the vapour of the fuel is constant except at pressure levels close to the upper limit of the pump. At the latter pressure level the vapour stream will be re-directed into the high vacuum space. In the case of the ejector pump the vapour pressure of the pumping fluid is higher. Thus, if operated in the low gas pressure region, the vapour stream leaving the jet will hit the walls of the mixing area at a large angle causing shock waves which will re-direct the vapour into the high vacuum space. If operated at medium pressures, the vapour cone in front of the jet will be constricted and extended so that the gas entrained by the

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outer layers of the vapour stream will reach the area where the fore vacuum pump becomes effective. But, if the pressure on the vacuum side is still higher, the vapour cone will be so severely constricted that the vapour stream will nowhere touch the walls of the mixing area. As a result back diffusion from the fore vacuum side will take place. This explains the pointed shape of the speed-pressure curve of the ejector pump. Similar deductions are made on the subject of ultimate vacuum obtainable and fore vacuum requirements.

Sommaire : On a étudié le mécanisme de pompage des pompes à diffusion et à éjecteur.

Article by R. Jaeckel H. G. Nöller & H. Kutscher *Vakuum-Tech.* 3, April 1954 1-15

Mechanical Production of Expiratory Rates Surpassing the Capacity of Human Coughing 140/I

See Abstract No. : 192/I

Economical Use of a Leak Detector Spectrometer 141/I

France. Leak detection problems in vacuum technique are discussed thoroughly There are two types of leaks: The real leak and the virtual leak. The latter is usually due to degassing phenomena in the receptacle pumped. It can normally be traced with the help of a liquid nitrogen cold trap. There are various methods of detecting the position and size of real leaks. The sensitivity of these methods depends on the nature of the probe material used and on the sensitivity of the detection means employed. At pressures above 10^{-2} mm. Hg alcohol can be used as the probe fluid and a discharge tube as the detector. Another method useful at lower pressures is the use of carbon gases as the probe gas and an ionisation gauge, coupled with a liquid nitrogen trap, as the detector. The carbon gas, having penetrated into the receptacle is condensed in the liquid nitrogen trap and as a result the gauge indicates suddenly a lower pressure. The most important method discussed in this report is the mass spectrometer method. In theory any gas can be used as the probe gas. In practice helium is the most suitable because its molecular mass is small, it is never present to any great extent in degassing products and the helium content of air is very small. The mass spectrometer can detect a minimum helium concentration of 1:400,000 (γ_o) in air. The size of the leak can be calculated from

$$f = \frac{V}{H}\frac{\triangle p}{\triangle t}$$

where f is the throughput of the leak, V is the volume of the vessel pumped, H the pressure in the vessel and $\triangle p/\triangle t$ the pressure rise due to the leak. In order to neutralise the effects of a leak of given size the pumping speed must obey the equation

$$Hf = Sp$$

The time lag incurred before the spectrometer gives an indication of the presence of a leak is given by the formula

$$t_a = \frac{V}{S}\log_e\frac{1}{1-\frac{SP\gamma_o}{Hf}}$$

and the minimum leak size detectable by the apparatus is given by

$$f_{min} \geqq \frac{SP\gamma_o}{H}$$

The period required for the signal indicated in the spectrometer to disappear is longer than the time lag t_a. In order to reduce this dead time to a minimum it is advised to stop supplying helium to the leak as soon as it is indicated in the spectrometer. The formulae given apply only to simple leaks, not to the type which results from faulty welding, for instance, which include air pockets, etc. Also, the mean free path in the receptacle pumped should be larger than the size of the receptacle. Five different modes of applying the mass spectrometer for leak detection purposes are described. Where the receptacle pumped has a volume varying from 2.5 to 17.5 litres a rotary pump only is required. Where the receptacle tested is only of a few cubic centimetres volume no special vacuum pump at all is required, the pumping action of the spectrometer itself is sufficient.

Sommaire : Discussion des principes de base de la détection des fuites et références spéçiales sur l'emploi du spectromètre de masse avec l'hélium comme gaz de détection.

Report by R. Geller *C.E.A. Rep. No.* 256 April 1954

Experiments in the Design of Ceramic Electron Tubes 142/I

See Abstract No. : 116/II

Results of Experiments on the Behaviour of Nickel for Cathodes Heated Under Vacuum 143/I

France. In the course of work generally aimed at improving the performance of thermionic valves, degassing phenomena in nickel, when heated under a high vacuum, were studied. Gas can be taken up by a solid in three different ways: (1) By chemical reaction, (2) by adsorption on the surface and (3) by absorption resulting from diffusion into the bulk of the material. Only by melting can the total amount of gas held by metal be liberated. As it was the object of the studies to simulate operational conditions in a radio valve during the tests, the heat treatment was carried out at about 1,000°C., *i.e.* in the solid state. The heat source was high frequency. The vacuum in the vessel was about 10^{-5} to 10^{-6} mm. Hg. Evacuation was carried out by diffusion pump. A high

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fore pressure was employed to avoid recombination of the gas with the metal. The gas evolved was collected in a receptacle of known volume and the volume of the total gas mixture collected was determined by pressure measurement. Details are given of the method of analysis employed. The total amount of gas collected in one experimental run was approximately 50 mm.³ N.T.P. and the sensitivity of the analytical procedure approximately 0.5 mm.³. The experimental results are given in tabular and graphical form. In one case nickel samples 0.5 mm. thick ready for drawing were heated in vacuum at 970°C. for 1 hour. Subsequently the nickel samples were annealed for 15 minutes in a cracked-ammonia atmosphere. The results are shown in the table reproduced below.

Gas	*Nickel*	
	Before Annealing in a cracked gas atmosphere at 800° C and 15 mm. Hg	*After* mm.³/g. N.T.P.
Total amount	115	76
Oxygen	5	0
Hydrogen	21	30
Carbon monoxide	64	26.5

Experiments with drawn cathode tubes indicated increased oxidation, possibly as a result of the drawing process, and the remedial influence of annealing under a reducing atmosphere. Generally, nickel with a low hydrogen content heat-treated at 970°C. shows a lower rate of degassing than nickel with a high hydrogen content. Also, heat treatment in vacuum will reduce the total gas content appreciably but additional treatment in a reducing atmosphere is often desirable. Finally, processing of the nickel in atmosphere after vacuum treatment will lead to appreciable re-absorption of gases. Metal deposits formed by evaporation in valves may adversely affect insulation and emission. Methodic experiments carried out by the author to investigate the behaviour of nickel in this respect showed that very little nickel evaporates under the operational conditions of the valve. But in experiments on tubes fitted with drawn cathodes three different kinds of deposits were noticed consisting of (*a*) magnesium, (*b*) a mixture of copper, tin and nickel and (*c*) residual material of an organic character most likely introduced originally during the drawing process. The amount of magnesium deposited was equivalent to 0.1% of the weight of the nickel processed but deposition was restricted to the first ten minutes of the treatment, when it ceased abruptly.

Sommaire : Les effets de dégazage du nickel du type employé dans les valves thermioniques, ont été étudiés, dans les conditions semblables à celles d'une valve en functionnement.

Article by J. Richard *Le Vide* 9, May 1954 28-32

144/I

Reactions Between Oxide Cathodes and Gases at Very Low Pressures

United Kingdom. The quality of thermionic emission from oxide-coated cathodes may be appreciably affected by chemical reactions between the cathode material and residual gases. Experiments aiming at exploring these conditions should be carried out at the operational pressure of thermionic valves and at the operational temperature of oxide cathodes. Due to the latter pulsed currents should be employed for measuring the emission. The author reports on an investigation carried out on these lines employing an experimental set-up which represents a development of the capillary method used essentially in the past for the assessment of getter performance. The experimental 'tube' was fitted at one end with the oxide cathode, 2 grids and anode, and close to it at the other end, with the ion collector of the ionisation gauge and a suppressor cylinder for the suppression of photoelectrons originating from the collector. This facilitated the use of the space-charge limited emission currents drawn simultaneously with the saturated pulse emission for pressure measurement. The tube was connected by means of a capillary of 4 cm. length and 2 mm. diameter to the manifold of a diffusion pump. Gas could be admitted *via* a needle valve into the manifold. Thus the rate at which gas was admitted to the cathode could be calculated from the pressure in the manifold, the pressure in the tube and the conductance of the capillary. Details of the electronic circuit used are given. The oxide cathodes were coated with barium and strontium carbonates in equimolar proportions. The cores were either nickel or platinum. The effects of oxygen, carbon dioxide, hydrogen, nitrogen, methane and water vapour on the performance of the oxide cathodes were investigated. In the case of oxygen the poisoning equilibrium was found to be unaffected by the material of the cathode core. Gas flow measurements indicated that the oxygen is taken up by active barium responsible for the emission as seen in the table reproduced below.

Rate of Consumption of Barium during Poisoning
(*Oxygen*)

$T_c = 1075° K.$ Ni-Core $F = 17.5$ cm.³ sec.⁻¹				
Time (min.)	P_{man} (mm. $\times 10^{-6}$)	P_t(mm. $\times 10^{-6}$)	I_p (A)	R_{Ba} ($10^{-4}\mu g.\ sec.^{-1}$)
—			2.45	
½	3.2	0.85	1.90	6.0
2	3.2	1.0	1.48	5.6
4	3.3	1.2	1.15	5.4
7	2.55	1.3	1.02	3.2
10	2.1	1.35	1.00	1.9
14	2.0	1.25	1.00	1.9

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Comparing the ion current with the rate, at which excess barium is consumed, indicates that poisoning is not effected by ions but by neutral particles. Further, it is indicated that only 1% of the molecules reaching the cathode surface reacts with the barium. This and other observations suggest that the greater part of the oxygen diffuses into the interior of the cathode. Finally, in the temperature range of 1,000 to 1,100°K, the association of oxygen and barium is loose enough to permit complete reversibility of the process but in the temperature range 1,200°K and over the association seems to be permanent and no substantial recovery of emission can be obtained. Poisoning by carbon dioxide is more pronounced than by oxygen in the pressure range below 3×10^{-7} and less severe in the pressure range above 3×10^{-7} mm. Hg. Also, the material of the core plays an important part in the process. Reduction of emission in the presence of nitrogen never exceeded more than 5% at the pressure level of 10^{-5} mm. Hg. The effect of water vapour depends on the pressure. Up to 10^{-5} mm. Hg water vapour increases the emission but at pressures above 10^{-5} mm. Hg water vapour has a poisoning effect. Similarly methane is activating at very low pressures and poisonous above 5×10^{-5} mm. Hg. An explanation of this phenomenon is given. The author concludes with remarks on the effects of excess barium on the emission characteristics.

Sommaire : Les effets d'empoisonnement ou d'activation sur les cathodes à oxyde par des gaz présents dans les valves thermioniques ont été etudiés à l'aide d'une technique semblable à celle employee pour l'etude des getters.

Article by S. Wagener *Proc. Phys. Soc.* **67B**, May 1954 369-386

O

Influence of Electronic Impact on the Rate of Sorption of Gases onto Getter Materials

145/I

United Kingdom. Observations by previous workers which suggested that magnesium getters some gases more rapidly if the gas molecules are ionised by electron impact has led to the view that this is a phenomenon applying to gases in general and other getter materials. The author reports an experiment carried out with the aim of confirming these observations. Barium was the getter material tested. A Knudsen gauge was used for the pressure measurement thus avoiding the electron impact on the gas which occurs when an ionisation gauge is employed. Instead a controlled electron impact was provided by placing a diode with a tungsten cathode and a nickel anode in a side tube connected to the gauge. The anode voltage of the diode was 100 V and the electron current about 10 mA. The gettering rates measured are shown in a table partly reproduced below.

Influence of Electron Impact on Gettering-Rates (in cm.³/sec.)

Getter	*Gas*	*Getter area (cm.²)*	*Number of measure-ments*	*Average value of gettering-rate*	
				without impact	*with impact*
Barium	Oxygen	6	7	1,200	1,250
"	Carbon monoxide	6	5	1,800	1,800
Magnesium	Carbon monoxide	30	2	7	12

O

The results, carefully checked to make sure that the high gettering rates obtained are not due to any electric or electronic phenomenon, contradict the previous theory.

Sommaire : On rapporte des expériences prouvant que les effets d'ionisation n'agissent pas sur les performances des getters.

Letter by S. Wagener *Nature* **173**, 10.4.1954 681-685

Improvement i.o.r.t. Active Getter Material

146/I

United Kingdom. It is known that vaporising barium in the presence of an activating gas improves the activity of the barium getter. However, this method has the disadvantage that the activating gas has a comparatively high equilibrium pressure at the end of the process. To overcome this difficulty it is proposed to produce active getter material as follows: Commerical barium is melted in a rare gas atmosphere, for instance argon. For 20 minutes a stream of argon containing 8% nitrogen is passed through the melt. On cooling down, the barium will contain approximately 1% by weight of nitrogen. The composition of the gas retained in the barium will have changed to about 75% by volume of nitrogen and only 25% by volume of hydrogen. This quantity of hydrogen is sufficient to activate the barium when the getter is fired. At the same time, the hydrogen equilibrium pressure established in the component after firing the getter is low enough to ensure the absorption of any quantity of hydrogen present in the component's atmosphere.

Sommaire : Description d'une méthode de préparation d'un matériau getter actif, réduisant sa contenance en gaz et ainsi augmentant son efficacité.

Philips Electrical Industries Ltd. *Brit. Pat.* 710,247

Evacuating Photographic Materials for Use in Electron Optical Apparatus

147/I

See Abstract No. : 110/II

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Principles of Vacuum Distillation of Metal Mixtures

148/I

See Abstract No. : 195/I

Evaporated Metal Films

149/I

Australia. A comprehensive review is given of the work on the production (Part I) and properties (Part II) of evaporated metal films published in the period 1927 to 1953. The first part gives information on the different methods employed, the materials used in the process and the various types of evaporation plant available. A list of fifty different metals is given showing the temperature at which their vapour pressure is 10^{-2} mm. Hg, also, a table setting out filament materials (evaporation sources) for the evaporation of various metals in order of merit, reproduced below.

Filaments for the Evaporation of Various Metals in Order of Merit

Metal	*Filaments*	*Metal*	*Filaments*
Ag	Ta Mo Cb Fe	Ni	W
Al	W Ta Mo Cb	Pb	Fe Ni Chromel
Au	W Mo	Pt	W
Ba	W Ta Mo Cb	Sb	Chromel Ta W
Be	Ta W Mo	Se	Chromel Fe Mo
Bi	Chromel Ta W	Sn	Chromel
Cd	Chromel Cb Ta	Sr	W Ta Mo Cb
Co	Cb	Te	W Ta Mo Cb
Cu	Cb Mo Ta	Th	Mo
Fe	W	Ti	W Ta
Ge	Ta Mo	Tl	Ni Fe Cb Ta
Mg	W Ta Mo Cb	V	W Mo
Mn	W Ta Mo Cb	Zn	W Ta Mo Cb

The second part, which forms the bulk of the paper, deals with the methods of measuring film thickness covering both optical and gravimetric methods. It is pointed out that a comparison of the results of the two methods indicates a lower density of the film than that of the respective bulk metal in many cases. The anomalous electrical properties of thin films are assumed to be due partly to the granular nature of the structure and partly to the limitation of the mean free path of the conduction electrons. Films of several thousand Ångstroem thickness approach the properties of the bulk material. Films of the thickness less than 100 Å are obviously influenced in their electrical performance by the nature of the substrate and the gas environment. The results of various investigations into the magnetic properties of thin films are reported. Some data is given on the photoelectric properties of thin films of certain materials, such as bismuth, sodium, rubidium and caesium. In the section on optical properties the close relationship between optical and electrical performance of the films is emphasised. The crystallography of thin films is discussed in great detail. Reference is made to the fact that the lattice constants of thin films may vary according to the nature of the substrate. Finally, the merits of evaporated metal films facilitating the exploration and exploitation of surface phenomena such as adsorption and catalysis as well as surface reactions in general are reported. In this connection the B.E.T. method of measuring surface area using krypton, methane and butane is mentioned. The mass spectrometer has been applied to the study of catalytic reactions at a metal surface. The gas sorption and reaction characteristics of certain evaporated metal film have led to their use as getters in thermionic valves. It has been shown that both speed and capacity in contact-gettering of a given gas is generally increased in the order of magnesium, calcium, bright barium and black barium. A method of joint discharge- and contact-gettering has been applied with success. In his conclusions the author points out that the method of preparation and substrate properties greatly determine the film structure. Strict methods of preparation are required to produce films suitable for accurate and absolute measurement of fundamental properties. 381 References.

Sommaire : Un article sur les couches minces de métal, passant en revue tous les travaux publiés dans ce domaine de 1927 à 1953.

Article by J. A. Allen
Rev. Pure Appl. Chem.
4, June 1954
133-170

How to Finish Metal Products by Vacuum Metallising

150/I

United States. The motor car industry, the fancy hardware industry, the toy industry and the light fixture industry are among the important users of vacuum metallisation. The present paper gives a general survey of the application of the vacuum evaporation technique to the coating of metal die castings or steel stampings. The components are cleaned and then lightly phosphated for the dual purpose of rust protection and providing a good bonding surface. Subsequently the component is dipped in, or sprayed with, a lacquer of the synthetic resin type which is cured by baking at 300°F for about 20 minutes. This seals off any pores in the surface of the component and thus reduces the pumping-down period and at the same time ensures a high gloss surface in the finished product. For actual metallisation a number of components are loaded on racks, which in the case of complicated surface contours are designed to rotate during the operation, and are transferred to the vacuum chamber. The chamber is evacuated by a pumping unit consisting of a mechanical pump, a booster pump and a diffusion pump. Operational pressure is 0.5 micron Hg or less which should be obtained in about 15 minutes. In the early stages of pumping-down the filaments (evaporation sources) are switched on to remove moisture

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and to allow for expansion. When a pressure of 1 micron Hg is reached the temperature of the filament is brought up to 1,200°F which is the melting point of aluminium, the most commonly used coating material. In the molten state the aluminium will spread over the whole length of the filament and, in addition, act as a getter. At operational pressure the filament temperature is rapidly raised to 1,800°F and the aluminium evaporates. Evaporation takes between 5 and 10 seconds. If properly treated, filaments should last up to 15 cycles. The finished articles are given a protective top coat of a suitable lacquer about 2 to 3 mil thick which can be dyed to give any desired colour tint. Besides aluminium, gold and silver can be readily evaporated by the same procedure. Nickel and chromium, however, attack tungsten and for that reason are electroplated to the filaments prior to the evaporation process. The effectiveness of the protective top coat of lacquer is indicated by the fact that vacuum metallised surfaces on zinc diecastings have been exposed for 800 hours to salt spray without deterioration.

Sommaire : Un article décrivant les méthodes employées pour la métallisation de metaux et les applications de cette technique.

Article by J. G. Seiter *Prod. Finish.* May 1954

Notes on Solution of Problems in Odd Job Vapour Coating

151/I

United States. Nomographs and other graphical aids are presented as a means to economic vacuum coating in odd job work. The author considers first the mathematics of the basic conditions in evaporation: (*a*) The evaporation source is a small solid sphere and the surface to be coated is a hollow sphere with the evaporation source in the centre. Assuming the evaporation source is a droplet of molten metal, originally 1 cm. long and *d* mm. in diameter and expressing the deposit thickness in *T* Å and the distance between source and surface by *r* in cm. the thickness of the deposit obtained is expressed by

$$T^{\circ} = \frac{40.3d^2l}{r^2} \qquad (1)$$

(*b*) The evaporation source is a small sphere as before but the surface to be coated is plane and situated at a distance *r* from the sphere. At the foot of the perpendicular (=*r*) the thickness obeys the above equation but at a point on the surface, *x* distant from the foot of the perpendicular, the metal lands on the surface obliquely and consequently must cover a greater area. As a result the deposit is thinner by the ratio cos θ to 1 where θ is the angle formed by the perpendicular at *x* and a straight line connecting with the sphere. The thickness of the deposit at this point is expressed by

$$T^{\circ} = \frac{40.3d^2l}{r^3} \, f \qquad (2)$$

Where

$$f = \frac{r^2 \cos\theta}{r^2+x^2} = \left[1 + \frac{x^2}{r^2}\right]^{-\frac{3}{2}}$$

The author demonstrates that Equ. 1 can equally well be applied to the condition of a linear evaporation source as long as the surface to be coated is situated on the axis of the cylinder whose length is half its diameter or, alternatively, the surface is placed parallel to the length of the cylinder whose length is 1.8 times its diameter. Both these conditions can be obtained in practice by choosing a suitable form of evaporation source. In the above considerations it is assumed that the molecular rays travel on straight lines. At a pressure of 10^{-4} mm. Hg the mean free path in air is 70 cm. Thus, for a path length of 7 cm. only 9% of the metal atoms in flight will suffer collisions. Normally operational pressures in evaporation processes are somewhat below that figure. The author proceeds to discuss filament design and physical properties of the metals to be evaporated. The filament material considered is tungsten. To facilitate quick determination of suitable design data for tungsten filaments monographs are given for lead length allowance, wire diameter—temperature—resistance wire diameter—current—temperature and wire diameter—length. The melting point of the metal to be evaporated is important. High melting point materials will require high filament temperatures. High filament temperatures will cause excessive heat radiation to the surface to be coated. For this reason such material should be evaporated by short flashes of the filament, allowing for a cooling period between each flash. At 2,000°K. silver evaporates 200,000 times faster than platinum but even at 3,000°K. platinum evaporates 20,000 times faster than tungsten. This gives an indication of the amount of tungsten which can be expected in the deposit as an impurity. In the final section of the article the structure of thin films is discussed and data is given on the relationship between electrical and optical properties of thin films.

Sommaire : Présentation de graphiques facilitant l'opération d'appareil de déposition sous vide pour des travaux particuliers differents, de ceux d'une production en masse.

Article by W. L. Bond *J. Opt. Soc. Amer.* 44, June 1954 429-438

Virus Vaccines Against Rinderpest: The Rabbit Rinderpest Virus. Part II

152/I

See Abstract No. : 49/IV

Lyophilisation of Poliomyelitis Virus, Heat Inactivation of Dry MEF1 Virus

153/I

See Abstract No. : 50/IV

Curbing Jack Frost

154/I

See Abstract No. : 151/III

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13 — ELECTRICAL SCIENCES — 13

Electronic Controlled Pumping System 155/I

See Abstract No. : 95/II

Ionisation and Excitation of Mercury Vapour by Positive Ion Impact 156/I

Pakistan. Townsend's postulate that atoms of a gas can be ionised directly by the impact of positive ions is still the subject of controversy. The authors report on experiments supporting Townsend s assumption. Mercury vapour was bombarded with potassium ions. Ionisation by electrons was eliminated by the provision of electron traps. Radiation resulting from ionisation was recorded by a Geiger counter with a highly photo-sensitive cathode, the instruments being shielded effectively against short ultra-violet radiation emitted by the ion source. The radiations recorded were proved to come from the mercury by freezing out the vapour. The results are shown in a graph. Counts were first detected at a bombarding energy of about 220 eV and the intensity was found to increase monotonically with energy. In view of Jones' results indicating that potassium-ion bombardment produces little change in intensity in the energy range of 300–800 eV it is concluded that the results of the present experiments represent mainly recombination spectra following ionisation of the mercury atoms by ion impact.

Letter by R. M. Chaudhri C. M. Aslam & S. U. Hasan *Nature* 173, 19.6.1954 1186

Sommaire : Des expériençes, faites sur la vapeur de mercure, montrent que l'ionisation est obtenue directement par chocs d'ions positifs, confirmant ainsi un postulat emis auparavant par Townsend.

An Ionisation Manometer and Control Unit for Extremely Low Pressures 157/I

See Abstract No. : 103/II

Thermistor McLeod Gauge for a Pressure Range $1\text{-}10^{-7}$ mm. of Mercury 158/I

See Abstract No. : 99/II

An Investigation on Aluminium Electrolytic Capacitors and the Development of a New Type of Aluminium Oxide Capacitor 159/I

United Kingdom. The performance of electrolytic capacitors of the aluminium oxide type is limited by their temperature characteristics. An investigation is reported aiming at an extension of the working temperature range of this type of capacitor. Part I gives the results of ageing tests of normal production-type capacitors of nominal capacitance of 8 μF and 16 μF lasting up to 10,000 hours and carried out at the rated voltage but at an elevated temperature, *i.e.* 70°C. Three groups of capacitors were tested: (*a*) Units made from etched 99.8%—purity aluminium foil impregnated with ethylene glycol-boric acid electrolyte. Rated voltage 500V. (*b*) Units of similar design but impregnated with an electrolyte having improved performance at low temperatures, rated voltage 350V, and (*c*) units of a special design, rated voltage 375V. In all tests leakage current, capacitance, power factor and loss of weight were determined. The results of the measurements are shown in graphs. Group (*c*), although tested at 85°C, showed better results than Group (*a*) and (*b*). The leakage current dropped to 40 μA after 4 hours, a value obtained by (*a*) only after 2,000 hours. The capacitance fell initially approximately 3% but after 1,100 hours it increased again at an average rate of about 1% per 1,000 hours. In the case of (*a*) the capacitance decreased continuously throughout the tests. The power factor of (*c*) was 1.5% after 3,000 hours whereas in the case of (*a*) it was 2% all through the test. The continuous reduction of the capacitance indicates progressive drying out of the electrolyte. In addition, electrolyte is lost through the vent necessary to extract the hydrogen evolved electrolytically at the cathode due to the action of the leakage current. In an 8 μF capacitor hydrogen was found to be evolved at a rate of 0.097 ml. p. coulomb. Hydrogen evolution can be counteracted with some success by adding a known hydrogen acceptor such as azobenzene to the electrolyte: 6 mg. of azobenzene in an 8 μF capacitor will accept 8 cc. of hydrogen. The ageing tests proved that the leakage current and the electrolyte are the main causes of failure. Leakage current would be reduced if the aluminium oxide film could be improved. A fundamental study of the forming process and properties of the oxide layer is reported in Part II. It is essentially concerned with oxide layers formed anodically in boric acid, adding sodium borate where necessary in order to increase the conductivity of the electrolyte. It covers methods of estimating the thickness of the oxide layer, effects of voltage, temperature and processing time on oxide thickness and various aspects of gas evolution at the electrodes. Not all oxygen ions migrating to the anode unite with the aluminium ions. Some free oxygen in the form of gas bubbles is liberated continuously, presumably as a result of oxygen ions contacting impurities in the aluminium. Certain characteristics of the gas evolution suggested the presence of cavities probably located at impurity centres in the aluminium surface and the degree at which such defects are present governs the magnitude of the leakage current during the operation of the capacitor. In order to eliminate effectively both major causes of failure an attempt was made to develop a dry capacitor of the aluminium oxide type. This is reported in Part III. In the new design an oxide layer is formed on high purity aluminium by electrolysis at 500V in a boric acid solution. The anodised foil is then immersed in a 0.5% solution of Araldite in acetone and allowed to dry slowly in acetone vapour. The Araldite coating is introduced to counteract 'fault clearing' in the base film with the attendant loss of capacitance. After baking the foil at

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200°C for 40 minutes in order to polymerise the resin, an aluminium layer of about 10^{-5} cm. thickness is vacuum-evaporated onto the foil at a pressure of 10^{-5} mm. Hg. The following table gives the performance characteristics of small oxide Araldite dielectric capacitors of approximately 15 cm.² area dried *in vacuo* instead of in air.

Conditions of Test	*Oxide + Araldite*		
	Ohm-Farads at 90 V	*Capacitance* 10^{-9} *F/cm.²*	*P.F. %*
Ordinary atmosphere	163 (B) 56 (M_8)	—	—
Dry at room temperature	4,000 (B) 1,400 (M_{11})	7.6	0.6
70°C	175 (B) 90 (M_9)	7.7	0.75

In the ohm-farads column the letters after the figures indicate the best value (B) and the mean value (M_x) of x samples.

Sommaire : Des essais de vieillissement faits sur des condensateurs électrolytiques à l'aluminium ont permis de trouver les principales causes de panne, et un nouveau type de condensateur à oxyde d'aluminium a été developpé.

Report by G. C. Robinson *E.R.A. Rep. No. L/T* 305 June 1954

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Improvements in Metal-Coated Recording Strips

160/I

Germany. Metal-coated strips of the type employed in instrument recorders, fitted with an electric stylus which burns a trace into the metal coating of the strip, have in the past been manufactured by evaporating the metal-coating onto a paper base directly, see Vol. I Abstract No. 64/I. Experience has shown that chemical action attributed to water in the base causes corrosion of the metal-coating, and in order to overcome this difficulty it is proposed that the metal should be deposited on a water-free, non-hygroscopic and chemically neutral surface. This surface can be part of the backing itself, *e.g.* polystyrene, or can be a film of varnish applied to the paper backing. In the latter case it is advisable to varnish both sides of the paper to prevent damage of the metallised top surface when the strip is rolled for storage. Cadmium has been found to give the most stable metal coating. Recommended layer thicknesses are 15–20 μ for the paper backing, 0.8–1 μ for the varnish film and 0.05–0.1 μ for the metal coating.

Sommaire : On discute les mesures prises pour éviter la corrosion des feuilles métallisées, d'enregistreur.

Patent by R. Bosch G.m.b.H. *Brit. Pat.* 708,912

Aluminising T.V. Tubes

161/I

See Abstract No.: 105/III

14 — MECHANICAL SCIENCES — 14

Design of a Large Vacuum Vessel

162/I

United States. In connection with the construction of a large linear accelerator for the A.E.C. the erection of a horizontal cylindrical vacuum vessel was required of the dimensions 60 ft. diameter and 87 ft. length to house a resonant cavity operating at radio frequency. The vessel was to operate at a pressure of 10^{-6} mm. Hg produced by 48 diffusion pumps of 32 inch. diameter, each of which was connected separately to the vessel *via* a 24-inch. diameter vacuum valve. The two heads of the cylindrical vessel had to be designed to permit the installation of other equipment close by, outside the vessel, and one head had to have a door of 20 ft. diameter to admit a car for the transportation of heavy equipment. Some guidance in the design of the vessel was given by the ASME Unfired Pressure Vessel Code but many items such as the methods of support had to be developed independently as the code deals only with vacuum vessels erected vertically. The construction of the vessel was entirely by welding. The plate chosen was 0.5 inch thick steel (ASTMA 201 grade B). The small thickness of the plate was made possible by the provision of circular and longitudinal stiffeners. The longitudinal stiffeners were 19 inch. apart made from T beams, the T's being halves of a 12-inch. 31.8 lb. I-beam. The circular rings were spaced at 80 inch. and were 31½ inch. high. In addition there were longitudinal girders, 72 inch. apart, along the whole length of the top of the vessel. The end plates (heads) were concave. To allow for expansion in the axial direction the supporting columns were designed to permit slight bending in either direction. Special requirements for the welding of the vessel are discussed. Normal paints and lacquers could not be applied because they contain too many volatile components. Therefore protection against corrosion was effected by spray coating with aluminium by a special process. Loose particles of aluminium found after this operation were removed by peening with steel shot. Testing of the vessel was carried out in steps. In a preliminary test the pressure was gradually reduced to 0.5 p.s.i. During pumping-down, strain gauge measurements were made at 41 different locations. This test was followed by a load test simulating the weight of the internal equipment

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and using a total of 432 klbs. load. This was followed by a further vacuum test indicating that the structure is stiffer than originally estimated. Leak tightness of the shell was checked by two methods. In a preliminary test the halide leak detector was used and in a final test the helium mass spectrometer. In the case of the latter, leak testing of the vessel was carried out section by section. In that series about 20 leaks were found. None of these were in the region of the butt weld joints although there were about 3,000 ft. of such welds.

Sommaire : Description des parties mécaniques importantes d'une enceinte de 87 ft. de diamètre et 60 ft. de long travaillant à une pression de 10^{-6} mm. Hg.

Report by F. L. Maker *A.E.C. Rep. No. LRL-157* June 1954

Techniques Useful in Evacuating and Pressurising Metal Chambers 163/I

United States. Problems of making gas-tight joints in systems which are to be evacuated, baked out and then filled with a gas or, alternatively, kept under vacuum, have been solved by the authors using diffusion welding techniques. Standard valve parts were used in the fabrication of a semi-demountable flange. The base section of a 6L6 radio valve, having a silver-plated convolute, was bolted up against a copper plated steel ring on the header, using a 0.005 inch copper gasket between them. During baking, which was not carried above 375°C for fear of damaging the glass button in the valve part, the surfaces welded together. The seal was satisfactory for vacuum and pressures up to 300 lb. sq. inch and eight conductors were carried into the system by this means. The sealing of the pumping stem consisting of copper tubing of 5/16 inch o.d. was carried out by cold welding. A specially developed hand-operated pinch-off tool is described and illustrated. Copper parts were brazed together with an alloy of phosphorus and copper, known as 'Phoscopper', melting at 750°C. This eliminated the use of flux and reduced contamination in the system.

Sommaire : Discussion de méthodes simples permettant de faire des joints étanches aux gaz pour des appareils devant être cuits au four et de plus devant être ou remplis de gaz ou vide.

Note by H. V. Neher & A. R. Johnston *Rev. Sci. Instrum.* 25, May 1954 517-518

15 — PHYSICS — 15

Simple Linde Hydrogen and Helium Liquefiers 164/I

See Abstract No. : 131/II

The Nature of the Upper Atmosphere 165/I

United Kingdom. The following are the most important functions of the earth's atmosphere: (*a*) It acts as a thermal insulator, (*b*) it removes the lethal parts of the sun's ultra-violet light and (*c*) it causes meteors to volatilise. (*b*) and (*c*) take place at well defined regions of altitude, about 30 km and 100 km respectively, whereas (*a*) is the sum total of a number of thermal effects taking place over a wide range of altitudes. Exploration of the conditions in the upper atmosphere has been greatly helped by the recently developed rockets which can carry instruments nowadays up to a height of 200 km. There are a number of regions of altitude which have distinctly different compositions and properties. Above 12 km is the stratosphere which is mainly responsible for the absorption of primary cosmic rays. Between 20 and 40 km is the ozone layer which is responsible for the absorption of the sun's short ultra-violet light. Between 70 and 80 km is the sodium layer contributing to air glow effects. At 80 km is the first ionised region known as the D layer which can be the source of radio fade-outs. Up to 100 km the composition of the air is about the same as at ground level, *i.e.* 4/5th molecular nitrogen and 1/5th molecular oxygen. The pressure at that height is 10^{-7} of that on the earth's surface and the temperature is 240°K. Above this height the oxygen becomes predominantly atomic and a second ionised region, the E layer, occurs between 100 km and 120 km, important in connection with the performance of long-distance radio propagation. Two further ionised layers the F_1 and F_2 layers are present at 160 km height where the pressure is 3×10^{-9} of that at ground level and the temperature approximately 750°K. During the night the, F_1 and F_2 layers merge into the F layer which has a very much higher electron concentration than the E layer and is therefore even more important to long-distance radio propagation. Methods of directly measuring density, pressure and temperature by means of rockets at various heights are discussed. The speed of sound can be established from a determination of the ratio of the pressures measured by gauges attached to the sides of the rocket at different distances from the nose. This yields the Mach number of the flow past. Up to 100 km height direct measurements by rocket technique confirmed results obtained by indirect methods in the past. The author concludes with a discussion of the properties and functions of the ionised layers and remarks on the mechanism of the light emission phenomena, many of which arise at heights near 100 km.

Sommaire : Discussion sur la fonction et les propriétés physiques de l'atmosphère.

Article by H. S. W. Massey *Endeavour* 13, April 1954 81-85

A Sealed-Off Metal Proportional Counter for Cosmic-Ray Work 166/I

See Abstract No. : 55/IV

Particle Detectors of the Geiger-Muller Counter Type 167/I

See Abstract No. : 54/IV

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Abstract No. and References

The Continuous-Diffusion Chamber

168/I

Belgium. A brief survey is given of the developments in the design of cloud chambers beginning with the early types of the Wilson Chamber and ending with a description of the Langsdorf diffusion chamber. The traces obtained in the latter chamber are too weak to facilitate quantitative assessment in the study of nuclear events. The authors sought to improve the performance of this type of chamber and report on the design of a diffusion chamber developed by themselves. Its essential parts are a Pyrex cylinder, 15 cm. high and 23.5 cm. in diameter, closed at the upper and the lower end with copper plates. The upper plate is double-walled to facilitate the circulation of hot water which heats felt fixed to the inside surface of the copper plate. The vapour, supplied from outside, passes through the felt to the chamber. The bottom plate of the chamber is in contact with dry ice and kept at a temperature of –78°C. Supersaturation of the vapour occurs at a level of 3 cm. from the bottom of the chamber. The top plate carries a small Pyrex window which is kept free from condensation by blowing hot air on to it and which permits visual observation and photographic recording of the traces. A study was made of the design factors influencing the performance of the chamber. It appears that, with a diameter of 23.5 cm., the length of the chamber should not be iess than 10 cm. Further it was found that a vapour mixture of methyl alcohol (70%) and water (30%) gives the best results. The temperature gradient in the chamber should be about 7°C./cm. in order to obtain clear traces. Background interference can be minimised by applying intermittently an electrical field between the two cover plates. The quality of traces obtained in the chamber is demonstrated in an illustration showing a proton trace originating from the reaction Al^{27} (a, p) Si^{30}. The chamber has been operated at high pressure, atmospheric pressure and subatmospheric pressure. Clear traces of alpha and beta particles were obtained at a pressure of 8 cm. Hg.

Sommaire : On donne des détails sur le développement récent d'une chambre à diffusion continue travaillant d'une manière satisfaisante à pression réduite.

Article by M. R. R. Roy & M. L. Goes *Bull. Classe Sci.* **40**, June 1954 630-635

On the Simultaneous Determination of the Optical Constants and of the Thickness of Thin Absorbing Films

169/I

See Abstract No. : 110/III

The Optical Absorption Constant of Cadmium Oxide

170/I

See Abstract No. : 124/III

Method of Producing Graded Coatings

171/I

United States. A method is described of depositing coatings on glass or plastic surfaces by the evaporation technique which are of a predetermined graduated thickness. The effect is obtained with the help of stationary screens as distinct from moveable screens employed in the past for the purpose. The arrangement described assumes that the vacuum is high enough for the vaporised molecules to travel on a straight path and provides for the evaporation sources to consist either of one extended linear source or of several point sources in a line at equal distances from each other, the line being parallel to, and at an appreciable distance from, the surface to be coated. If the two ends of the linear evaporation source or the common axis of point sources are connected by a straight line with the respective opposite edges of the surface to be coated, the two (diagonal) lines will intersect at a point between the sources and the surface. A screen of a width equal to that of the surface to be coated and in a plane parallel to that surface is positioned in such a manner that its outer edge is located at the point of intersection of the two diagonal lines. Assuming that four point sources are used, positioned at equal distances from each other and symmetrical to a line perpendicular to the surface and passing through the aforementioned point of intersection, the path of the vapours originated in the four sources will be restricted in such a manner that four zones of equal width will be created on the surface which receive vapours (1) from all sources (2) from three sources only, (3) from two sources only, and (4) from one source only. There are various practical applications of this principle. Three examples are described. In one case a graded coating was applied to the top of a car windscreen of the dimension 14 × 30 inch., the graded coating to consist of a strip having a width of 3½ inch. and specified to be opaque at the upper edge. The coating was effected with the windscreen at a distance of 22 inch. from a tungsten filament which consisted of stranded wire coiled into a spiral over a length of 8 inch. The material to be evaporated (Inconel wire) was placed in the form of hooks on the spirals at 10 equally spaced points. The screen was at a distance of 15 inch. from the filament. The final position of its edge was determined by sighting along the hypothetical lines mentioned before. Operational pressure was 10^{-4} mm. Hg. The grading obtained was uniform as demonstrated in a graph showing the light transmission at various distances from the top of the windscreen.

Sommaire : Description d'une méthode permettant par évaporation sous vide d'obtenir un dépôt à gradation, en plaçant un écran entre la source et la surface dans une position permanente et correspondante à la géométrie desirée pour le dépôt.

D. W. Barkley & Libbey-Owens-Ford Glass Co. *U.S. Pat.* 2,676,114

Improvements i.o.r.t. the Production of Indicia on Solid Bases, Such as Cross-Wires

172/I

See Abstract No. : 113/III

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Abstract No. and References

Multilayer Dielectric Reflecting Films at Wavelengths between 4,000 and 4,600 Å

173/I

United Kingdom. The authors have studied the performance of multilayer dielectric interference filters in the wavelength range 4,000-4,600 Å. The values obtained from seven-layer reflecting films of cryolite and zinc sulphide on glass are given as reproduced below.

λ	4,100 Å	4,300 Å	4,500 Å
Reflection coefficient	0.956	0.958	0.960
Transmission coefficient	0.027	0.030	0.032
Absorption coefficient	0.017	0.012	0.008

These values, which have been revised since they were first published (*Nature*, **171**, 1953, 648), show that such films are superior to silver films when employed in Fabry-Perot interferometers at these wavelengths.

Letter by J. Ring & W. L. Wilcock *Nature* **173**, 22.5.1954 994

Sommaire: Données sur les performances de films diélectriques à sept couches, employés dans l'interferomètre de Fabry-Perot.

Improvements i.o.r.t. Electrodes for Photo-Electric Devices

174/I

Liechtenstein. The metal electrodes embodied in photo-electric devices such as blocking layer photo-electric elements must have good electric conductivity properties and high light transmission properties. Gold is the typical material used for such electrodes. It has a light transmission of 50%, a light absorption of 15% and shows a loss by reflection of 35%. A new method of preparing such electrodes is proposed which aims at an appreciable reduction, or complete elimination, of the loss by reflection. In the new method the metal part of the electrode is made from less expensive material such as copper or copper alloys, on to which a single layer or multi-layer film of dielectric material is deposited by vacuum evaporation or by a precipitation technique. The thickness of the single layer and the refractive index of the material deposited should be such that reflections occurring at the air—layer interface and those occurring at the layer—electrode interface should cancel each other out by interference. A suitable thickness is one quarter of the wavelength of the light frequency, the reflection of which it is intended to suppress. Multi-layer deposits built up from various dielectric materials can serve to suppress the reflection of light at a multitude of wavelengths within the spectrum of the incident beam. Suitable coating materials are metal oxides, such as silica and silicon monoxide and metal fluorides, sulphides and phosphides. As a result of the increased light transmission obtained by this method the metal electrode can be made thicker than the conventional electrodes. This benefits the electric conductivity properties of the electrode.

A. Vogt *Brit. Pat.* 708,449

Sommaire : On discute l'emploi de films, simples ou composés, de matériaux dielectriques dans les cellules photoelectriques.

Notes on Solution of Problems in Odd Job Vapour Coating

175/I

See Abstract No. : 151/I

Magnetic Behaviour of Thin Single-Crystal Nickel Films

176/I

United Kingdom. To overcome the uncertainties in prior determinations of the magnetic properties of polycrystalline thin films of ferromagnetic materials, the authors produced nickel films of a thickness range of 1,000Å–200Å on single crystal rocksalt substrates by epitaxial growth for electron diffraction, electron microscopy and magnetometer examination. The films were prepared directly in the electron diffraction camera by vacuum deposition on a (100) rocksalt face, so that their initial structure could be examined, and were afterwards removed from the substrate by dissolving the rocksalt. Annealing was carried out in vacuum at about 10^{-6} mm. Hg for one hour at 650°C. to remove twinning and strain. The film thickness was measured by transmission of mercury green light and the films were then submitted to magnetic measurement, first in a torsion magnetometer of the type normally employed for the determination of ferromagnet impurity, and subsequently in the magnetometer designed by Bate, Schofield and Sucksmith to locate the direction of magnetisation. It was found that the single-crystal films of nickel behave as a single domain structure. They magnetise spontaneously, the direction of magnetisation lying in the plane of the film. In the range of thicknesses handled by the authors, the coercivity decreases continously as the thickness increased from 200–1,000Å but the results indicate that the maximum coercivity lies just below 200Å and has the value – 350 OE.

Article by L. E. Collins & O. S. Heavens *Philos. Mag.* **45**, March 1954 283-289

Sommaire : Détails sur l'étude du comportement magnétique de films minces de nickel mono-cristallin.

Studies on the Creep Recovery and Annealing of Zinc Single Crystals

177/I

United States. On applying stress to a single zinc crystal beyond its elastic limit the resultant strain can be analysed to consist of the following components: Elastic strain (completely recoverable), initial plastic strain (partially recoverable), time-dependent transient creep and quasi-viscous creep where the rate of strain is constant with time. Under conditions of plastic strain repeated loading and unloading will result in the elastic

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limit being increased and the transient creep rate reduced under the same stress as in the previous test. This 'work hardening' effect can be overcome by annealing the crystal, whereupon the earlier elastic limit is restored and the original creep behaviour is reinstated. The investigation recorded here is concerned with the determination of the factors which influence such a recovery at 35°C. The zinc employed in the experiments was of 99.999% purity. The crucibles used in growing the crystals were of spectroscopically pure graphite. The graphite crucible holding a zinc rod in its $\frac{1}{4}$–inch diameter cavity was sealed into a glass tube which was evacuated to 0.01 micron Hg and then heated to a temperature above the melting point of the zinc, and the crystal was grown by slowly lowering the sealed tube through a sharp temperature gradient at a rate of 3.3 cm/hr. Details of the method for the determination of the crystal orientation are given. The means applied for loading the crystals are discussed. A pair of aluminium sleeves was attached to the crystal to determine the gauge length of the sample and to provide a means of fixing the extensometers which were employed to measure the strain. Details of equipment are given for annealing which was carried out at less than 1 mm. Hg pressure. Creep rates are expressed in terms of resolved shear strain rates and could be measured with a precision of about 1×10^{-6} minute^{-1}. 18 crystals were used in the experiments but due to the annealing effects many crystals could be used several times. Creep tests lasted from 60 to 300 minutes at resolved shear stresses of $1–2 \times 10^{-6}$ dynes/cm^2. The results of the experiment indicate (*a*) recovery of initial plastic strain through annealing is appreciably less complete than the recovery of transient creep. (*b*) In the quasi–viscous region transient creep cannot be recovered if the load is removed partially or completely for no more than two minutes, but allowing for an extended rest period of, say, 100 minutes with a rest load of 94% of the initial load or with zero load, transient creep can be partially recovered. (*c*) Under certain conditions recovery of quasi–viscous creep is obtained. In the discussion of the results the authors suggest that the mechanism underlying the creep and recovery phenomena observed may be explained in terms of the dislocation theory of plastic flow. In the final section of the paper the authors report on an unexpected phenomenon observed during the experiments, termed 'anneal hardening'. Zinc crystals annealed at a temperature above 200°C were found to be very much harder than crystals left to recover at room temperature. The higher the annealing temperature, the harder the crystal after treatment. No explanation for this behaviour could be found as the impurity content was less than 0.001 % and annealing took place in vacuum.

Sommaire : Etude étendue sur le phenomène de glissement des cristaux de zinc et sur leur comportement aprés recuit.

Article by M. Tanenbaum & W. Kauzmann *J. Appl. Phys.* **25**, April 1954 451-458

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Principles of Vacuum Distillation of Metal Mixtures 178/I

See Abstract No. : 195/I

16 — CHEMISTRY — 16

Pressure Limits of Flame Propagation of Propane—Air Mixtures 179/I

United States. The results of previous investigations into the low pressure limits of flame propagation were recorded in terms of pressure limit against fuel concentration. But there were indications that the values obtained may be governed by quenching effects produced by the walls of the chamber in which combustion took place. It was the object of the present investigation to clarify this relationship. The essential part of the experimental equipment used consisted of an all–glass test section comprising an ignition chamber and a propagation section. The latter was a tube 120 cm. long in each case but varying in diameter from 1.6 cm. to 6.6 cm. and was sealed to the ignition chamber which was 9 cm. diameter and 25 cm. long. Ignition was effected by a capacitance spark across a pair of electrodes introduced into the ignition chamber through the stem of a stopcock. A 47–litre volume connected to the test section served for the reduction of pressure disturbances during flame propagation. Evacuation down to 1 micron Hg was carried out by a diffusion pump backed by a mechanical pump. Details are given of the method of preparing the propane–air mixtures employed in the experiment. Mixtures containing more than 5% propane showed an erratic behaviour and for that reason were not recorded. After pumping down the propane–air mixture was admitted to the propagation section at the pressure at which the test was carried out and after equilibrium had been reached throughout the system, the mixture was ignited. Depending on the prevailing pressure the flame would either fill the whole of the propagation section or part of it, or alternatively would be extinguished in the area where the ignition section and propagation section joined. Pressure determinations were accurate within $\pm$ 1 mm. Hg. The measurements show that for a different concentration the minimum pressure for flame propagation rises as the diameter of the vessel is decreased. At a given pressure the concentration range of flammability is reduced when the tube diameter is reduced. Previous experiments have shown that, provided the vessel is large enough, concentration limits of flammability are independent of its diameter. In a log—log plot of the critical diameter for flame propagation *versus* pressure limit strict proportionality between the vessel diameter and the limit pressure raised to a negative exponent for each mixture is established. The authors found that their results could be correlated with those of Friedman and Johnston who measured propane—air mixture quenching distances by determining the narrow slit through which a Bunsen flame will flash back when mixture flow is reduced (See *J. Appl. Phys.*, **21**, 1950, 791–5). This is demonstrated in a table partly reproduced on the next page.

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Ratio of Critical Diameter to Minimum Slit Width

Propane in Air, vol. %	*Mixture Pressure, atm.*	*Critical Diameter, d_1, cm.*	*Minimum Slit Width d_2, cm.*	*Ratio d_1/d_2*
4.03	0.0300	6.6	4.18	1.58
	0.0473	4.7	2.80	1.68
	0.0555	3.8	2.44	1.56
	0.0684	2.8	2.03	1.38
	0.0882	2.2	1.64	1.34
	0.1397	1.6	1.10	1.46
				Av. 1.50

The results on the present experiments permit the conclusion that the value of the pressure limit for flame propagation is governed by quenching effects on the walls and does not depend exclusively on the composition of the fuel air mixture.

Sommaire : On a trouvé que la propagation d'une flamme dans une enceinte sous basse pression ne dépends pas exclusivement des propriétées du mélange gaz-air mais aussi de l'effet d'étranglement apporté par la paroi de l'enceinte.

Article by F. E. Belle D. M. Simon & R. C. Weast *Ind. Eng. Chem.* **46**, May 1954 1010-1013

Composition, Decontamination of Radioactive Gas Mixtures 180/I

See Abstract No. : 41/IV

The Adsorption of Gases on Nickel Oxide 181/I

United Kingdom. Nickel oxide prepared by heating nickel carbonate at high temperatures has the true stoichiometric composition of nickel oxide but nickel oxide prepared below 500°C. can hold excess oxygen in its structure, giving it semiconductor properties of the positive-hole type (cation vacancies). Oxides of the latter type can absorb oxygen and are catalysts for surface reactions involving nitrous oxide and carbon monoxide. The present work was undertaken to give data on the adsorption of gases in thin nickel oxide films existing as surface layers on nickel metal. The heats of adsorption of oxygen, carbon monoxide and carbon dioxide have been measured calorimetrically, and the catalytic oxidation of carbon monoxide has been studied. The experiments were carried out in a vacuum chamber pumped by a mercury diffusion pump which was capable of an ultimate pressure of 10^{-6} mm. Hg when used in conjunction with a liquid oxygen cold trap. A Garner-Veal calorimeter was used to measure the heats of adsorption. Three samples of pure nickel granules were obtained by heating nickel oxalate *in vacuo* with subsequent reduction in hydrogen. Nickel oxide was formed as a surface layer by saturation of the metal with oxygen at room temperature and gradual conversion to oxide at 300°C. Adsorption of oxygen was rapid, and the amount depended upon the film thickness and the original outgassing temperature. For a very thin film of 25 Å, almost a complete monolayer was adsorbed at 20°C, and after heat treatment at 400°C, another monolayer was adsorbed. These calculations were made on the assumption of an average number of Ni^{2+} ions per sq. cm. $= 10.97 \times 10^{14}$. Thicker (100 Å) films adsorbed very much less oxygen.. The initial heat of adsorption of oxygen was found to be 54 kcal/mole. This value was checked using nitrous oxide by measuring its heat of decomposition. Small quantities of carbon monoxide were absorbed, but carbon dioxide was adsorbed in similar quantities to oxygen. It was shown that the adsorption of carbon dioxide on an oxygenised site involved the formation of an adsorption complex with the approximate stoichiometric composition CO_3. In the discussion the mechanism of the adsorption process in nickel oxide is related to the properties of the material as a semiconductor.

Sommaire : On a étudié l'absorption de gaz sur des films d'oxyde de nickel et les résultats sout en corrélation avec les propriétés semi-conductrices du matériaux.

Article by R. M. Dell & F. S. Stone *Trans. Farad. Soc.* **50**, May 1954 501-510

Photolysis of Poly Methyl Vinyl Ketone: Formation of Block Polymers 182/I

United Kingdom. Both in the gas phase and in solution, the primary photolysis of aliphatic ketones has been shown to occur by two simultaneous reactions:

$$R, CO.R' \xrightarrow{h\nu} R. + R'CO. \rightarrow R. + R'. + CO$$

and

$$RCH_2CH_2CH_2COR' \xrightarrow{h\nu} RCH{:}\ CH_2 + CH_3COR'$$

It has now been verified that both these types of reaction occur in vinyl polymers containing a carbonyl group when irradiated. The fact that reactions of the first type occur with poly methyl vinyl ketone, in which free bonds are formed in the polymer chain suggested the possibility of forming block polymers, where a long chain of one type is attached to another long chain of a different chemical species. In a preliminary series of experiments poly methyl vinyl ketone was photolysed in the presence of acrylonitrile monomer with 3,130 Å radiation. The poly methyl vinyl ketone was prepared by polymerising pure monomer in dioxan solution, freeing it from volatiles subsequently by freezing and sublimation under vacuum. No precipitate formed when the acrylonitrile and poly methyl vinyl ketone were irradiated in dioxan solution, but when they were dissolved in

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each other a solid precipitate was obtained. After removal of the residual monomer by freezing and vacuum sublimation the precipitate was qualitatively fractionated into three components: (*a*) Consisting essentially of poly acrylonitrile, (*b*) the block polymer, representing about one third of the total and containing 75% acrylonitrile, (*c*) the usual degradation product of poly methyl vinyl ketone. The experiments appear to confirm that photolysis of poly methyl vinyl ketone in the presence of acrylonitrile is instrumental in the synthetic production of two further polymeric species—pure poly acrylonitrile and a block polymer of both materials.

Sommaire : On a trouvé que la photolyse du 'poly methyl vinyl ketone' en presence d'acrylonitrile conduit à la production de deux nouveaux genres de polymérique.

Article by J. E. Guillet & R. G. W. Norrish *Nature* **173**, 3.4.1954 625-627

The Combination of Methyl Radicals: Photolysis of Acetone at Low Pressures 183/I

Canada. The mechanism whereby methyl radicals, primarily produced by the decomposition of acetone exposed to ultra-violet radiation, combine to form ethane has been investigated before, down to acetone pressures of 20 mm. Hg. The present investigation extends this study down to a pressure level of 0.2 mm. Hg. The results obtained confirm the previously postulated third body requirements for ethane formation, acetone itself acting as third body. The experimental reaction vessel was a quartz cylinder 100 cm. long and 3.9 cm. in diameter. Heat insulation was provided by wrapping the cylinder with aluminium foil and Pyrex tape and the uniformity of the temperature within the cylinder was $\pm$ 1° over its whole length at 250°C. Heat was supplied by resistance heating. Tbe radiation was obtained from a B.T.H. ME/D 250 W. d.c. quartz window lamp which gives strong emission at 3,130, 3,660, 3,906 and 4,047 Å. To facilitate quantum yield measurements the line 3,130 Å emitted by the lamp was isolated. Light absorption was measured by means of a GL 935 photo-cell. Normally 5% conversion of the acetone was regarded as sufficient. All materials used in the reaction, *i.e.* reagent—grade acetone, acetaldehyde, benzene, perfluoromethyl*cyclo*hexane, perfluorobutane and carbon dioxide, the latter few representing added gases, were purified by distillation and outgassed prior to use. Separation and analysis of the reaction products was carried out by known methods and was checked with the aid of a mass spectrometer. Ethane was separated out in a liquid nitrogen trap (–210°C). Results for various acetone pressures are given in graphs. Adding the gases mentioned above increased the relative rate of ethane formation.

Sommaire : Etude du mécanisme de formation d'éthane à partir de radicaux de methyl produits par la photolyse d'acétone à basses pressions.

Article by R. E. Dodd & E. W. R. Steacie *Proc. Roy. Soc.* **223A**, May 1954 283-295

The Electron Microscopy of Photographic Grains. Specimen Preparation Techniques and Applications 184/I

See Abstract No. : 111/II

Evacuating Photographic Materials for Use in Electron Optical Apparatus 185/I

See Abstract No. : 110/II

An Improved Replica Technique for Electron Microscopy of Paint Films 186/I

United States. For the study of organic films, such as films of soft paints, it is essential to employ replica techniques which exclude the risk of exposing the film surface to high temperatures. The authors found that the silver-silica replication technique involves such a risk and report on a successful modification of that technique by substituting PVA (Elvanol, grade 51–05) for silver for the negative replicas. Care has to be taken that the negative is thin enough to obtain a high resolution and dissolves without injury to the silica. The PVA film is produced by flooding the specimen surface, draining off the excess and drying in a vertical position of the specimen for 8–16 hours. Longer drying periods result in brittleness. The PVA replica strips readily from paint film applied to tinplate and, while fastened to a metal holder with adhesive tape, is coated with silica by vacuum evaporation. It is desirable to prolong pumping at 10^{-4} mm. Hg up to 16 hours to eliminate all traces of absorbed water. 0.8 mg. to 1.4 mg. of silica are evaporated, the thickness of the silica layer depending on the roughness of the surface. The finished composite films are cut into one-eighth inch squares and placed, silica-side up, in distilled water, when the PVA dissolves away quickly. The silica replicas are then washed before transfer to the specimen grids. Shadowing of the replicas with chromium is recommended. The new method has been used for the study of the underside of enamel films. Examples of the application of the techniqnes to gloss enamels and lacquers are given in illustrations. The new method indicates pigment distribution and related defects most satisfactorily but finer details such as molecular configuration and small distortions of the paint vehicle are better shown by silver-silica replicas.

Sommaire : Description d'une technique de réplique double, modifiéé, pour remplacer la technique de réplique au silicate d'argent dans la réplique de surfaces molles de couche de peinture.

Article by A. S. Powell L. R. LeBras E. G. Bobalek, & W. Von Fischer *J. Appl. Phys.* **25**, June 1954 757-760

The Electron Microscope with Particular Reference to Its Use in Biological Research. I 187/I

See Abstract No. : 108/II

Preservation of Foods with Ionising Radiations 188/I

United Kingdom. A report is given of a short symposium held at Cambridge in February 1954 on the above subject. The powerful sources of ionising radiations that have recently become available open up possibilities

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of preserving food by killing the organisms that cause spoilage. Doses of $2–5 \times 10^4$ r.e.p. have been found to break the reproduction cycle of insects in foods (*e.g.* trichina larvae) and $1–5 \times 10^6$ r.e.p. are needed to inactivate virus. Many foods develop 'radiation' odours which may render them unsuitable for human consumption. This effect can be minimised by reducing the dose level to 2×10^4 r.e.p. which is still useful for disinfestation. Other measures are de-aeration, deep freezing, and, as an alternative to the latter, the addition of ascorbic acid. Each foodstuff has to be considered separately. Vegetables and fruit contain enzymes which are active during storage and are not normally inactivated by irradiation. The most serious biological effect of irradiation is a loss of the property of cell reproduction. Tissue cells show symptoms indicating tumour development. These adverse effects are attributed to the formation of free radicals in the presence of water. This aspect of irradiation effects requires further clarification. Available radiation sources are electrical generators supplying high speed electrons and radioactive sources producing beta or gamma rays. In the former class are Van de Graaff accelerators and travelling wave linear accelerators. With an electron beam power of 4 kW already obtainable from such generators material about 1 inch thick, if radiated from both sides, could be sterilised at a rate of about 1,000 lb. per hour at a cost of a few pence per lb. But as electrons have a comparatively low penetration the use of gamma rays may be preferable. These can be obtained either from spent reactor fuel rods during the cooling period or alternatively from fission products which remain after chemical processing of the fuel rods. A medium sized nuclear power station could sterilise about 1,000 tons per day from its spent fuel rods alone. The use of fission products has the advantage that the radiation sources would be transportable and it is expected that the cost will be about the same as in the case of sterilisation by electrical generators.

Conference Report by R. S. Hannan *Nature* **173**, 24.4.1954 752-754

Sommaire : Rapport sur un symposium de stérilisation de produits alimentaires au moyen de radiations ionisantes.

Physical Instability and Thermal Shock in Red Cells 189/I

See Abstract No. : 118/II

A Freeze Drying Apparatus for Neurohistochemical Work 190/I

United Kingdom. A freeze drying apparatus is described which employs no diffusion pumps or auxiliary heaters and is capable of drying simultaneously several groups of brain tissue in the form of slices for histochemical study of enzyme location.

(*Authors*)

Letter by D. Naido & O. E. Pratt *Vacuum* **4**, April 1954 195-197

Sommaire : Description d'un appareil à lyophiliser sans pompes à diffusion ou chauffages auxiliaires, capable de lyophiliser simultanément plusieurs grammes de tissues de cerveau, sous forme de tranches, pour localiser les enzymes durant l'étude histochimique.

Lyophilisation of Poliomyelitis Virus, Heat Inactivation of Dry MEF1 Virus 191/I

See Abstract No. : 50/IV

Mechanical Production of Expiratory Flow Rates Surpassing the Capacity of Human Coughing 192/I

United States. Various apparatus which produce an artificial cough in patients suffering from complaints which cause bronchial secretions and other impediments to normal respiratory functions (*i.e.* poliomyelitis, bronchial asthma, etc.) are already known but none of these designs employs negative pressure which would increase its effectiveness. Details are given of a new apparatus of this kind called E.W.N.P. (Exsufflation With Negative

Peak Expiratory Flow Rates During a Cough of Maximal Effort and During E.W.N.P.

Case No.	*Diagnosis*	*Cough cc./sec.*	*E.W.N.P. cc./sec.*	*E.W.N.P. × 100 Cough per cent.*
	A. NORMAL SUBJECTS			
1.		5380	7820	145
2.		8360	9040	108
3.		5380	7820	145
4.		10670	9040	85
5.		4570	6200	136
	B. PATIENTS WITH BRONCHIAL ASTHMA, PULMONARY EMPHYSEMA AND BRONCHIECTASIS			
1.	Pulmonary Emphysema	3000	7280	243
2.	Pulmonary Emphysema	3000	8910	297
3.	Bronchiectasis	7820	9450	121
4.	Pulmonary emphysema and Bronchial asthma	3000	6200	207
5.	Pulmonary emphysema	5790	8230	142

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Pressure). It consists essentially of a mask to be worn by the patient which connects to a pressure blower which gradually inflates the lungs of the patient to a pressure of about 30 mm. Hg above atmosphere after 2.5 seconds. At this stage either by manual operation or by relay action a valve shuts off the passage to the pressure blower and connects the mask with a suction blower. As a result the pressure in the line is reduced to 6 mm. Hg negative pressure within a period of 0.02 sec. and then more gradually further reduced to approximately –30 mm. Hg in a matter of seconds. The performance of the apparatus as compared with natural cough of normal subjects and patients is given in a table partly reproduced on previous page. The E.W.N.P. apparatus, tested on anaesthetised dogs, removed small solid objects as heavy as 0.6 g. and of 4.5 mm. diameter in the course of 6 to 12 exsufflations successfully.

Sommaire : Description d'un appareil récemment developpé produisant une toux artificielle chez les malades souffrant des poumons.

Article by A. L. Barach G. J. Beck & W. Smith *Amer. J. Med. Sci.* **226**, Sept. 1953 241-248

17 — METALLURGY — 17

High-Vacuum Fractionating Distillation Column 193/I

See Abstract No. : 143/III

Still 194/I

See Abstract No. : 132/III

Principles of Vacuum Distillation of Metal Mixtures 195/I

United States. The work described forms part of a study of the generation of power in breeder-type reactor systems. The recovery of generated fuel from a comparatively dilute mixture in breeder fuel, the recovery of unburnt nuclear fuel and the removal of fission products from the spent fuel constitute major items in the fuel processing connected with the operation of a breeder-type reactor. One of the requirements is the separation of plutonium from uranium. The author investigated the possibilities of employing the vacuum distillation method for this process in preference to a method involving chemical changes. The particular problem is that of 'differential' distillation and the greater part of the paper consists of a theoretical investigation into the mechanism of this process. Limitations of the usual simplified equilibrium and rate relations for the batch process are discussed, and relations of more general application are derived. The concept of relative volatility is used to express the vapour-liquid equilibrium relationships and the average processing rate if expressed in terms of a rate constant derived from the kinetic theory. A specific example is given to demonstrate the application of the theoretical conclusions arrived at: 500 lb. of a mixture is to be reduced in its plutonium content from 0.5 to 0.005% and the plutonium is to be obtained as an alloy with uranium containing no more than 15% of the latter. The uranium residue is to be reformed and returned to the blanket. If distillation is to be employed, a vacuum distillation furnace should be provided where the plutonium is stripped from the bulk of the uranium and in addition a smaller vacuum distillation furnace which would act as the enricher. Operation would be batch wise and operational temperature in the stripper furnace would be 1,730°C. The surface area necessary to perform the process in one day is: 5 sq. ft. for the stripper furnace and 21 sq. inch. for the enricher. A table gives additional values calculated for other operating conditions.

Sommaire : Etude essentiellement théorique sur l'emploi de la distillation différentielle sous vide, pour séparer le plutonium de l'uranium a l'état liquide.

Report by J. E. Vivian *A.E.C. Rep. No. LRL-88* March 1954

The System Uranium-Mercury 196/I

United Kingdom. An examination of the uranium-mercury alloy system, conducted as a part of a general investigation of uranium alloys, showed that, with the exception of alloys having very low uranium concentrations, all the uranium-mercury alloys were pyrophoric and have to be prepared and manipulated under vacuum. Glass tubes sealed by glass membranes were used as containers. Because of the high melting point of uranium and the low boiling point of mercury, alloys could not be formed by simple liquid-mixing. One method of preparation provided for clean uranium turnings to be placed in a Pyrex reaction tube with pure mercury in a sealed side arm. On passing hydrogen through the system and heating at 250°C for ½-hour, the uranium was converted to the hydride UH_3. The tube was then evacuated and the heating continued so that the hydride decomposed leaving uranium in the form of a grey pyrophoric powder. After cooling the tube was sealed off and the mercury was run into the powder from the side arm. Finally, the tube was kept in a furnace at 300°C for several hours to allow the contents to react. Specimens for X-ray and chemical analysis were obtained by joining an evacuated analysis receptacle fitted with X-ray and sample capillaries to the reaction tube. Powdered alloy was shaken into the capillaries, which were then sealed off and removed. The alloy system was examined over the whole composition range, and temperatures from –40° to 1,000°C. Determinations were made by X-ray techniques and by thermal analysis, microscopic examination being limited to mercury-rich alloys which must be examined at low temperatures. Electrical resistivity measurements and chemical analyses were also made. Thermal analysis was carried out in three temperature ranges, –60°C to room temperature, room temperature to 450°C and 450°C to 1000°C. A special type of Dewar flask was used for low-temperature analysis. Because of the

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high vapour pressure of mercury, experiments in the medium range were carried out with the sample tube inside a small steel container to minimise danger from tube burst. For the same reason, at high temperature, the specimen was placed in a small furnace positioned inside a gas-tight container, and nitrogen at 120 atm. was admitted to the container. The solid solubility of uranium in mercury and that of mercury in uranium were found to be small. The mercury appeared to have no effect on the transformation temperatures of uranium. Three compounds, UHg_2, UHg_3 and UHg_4 were identified and the crystal structures of UHg_2 and UHg_3 were confirmed but it was difficult to obtain a clear pattern of UHg_4.

Sommaire : On a étudié l'alliage mercure-uranium et plusieurs composés tels que UHg_2 et UHg_3 ont été identifiés.

Article by B. R. T. Frost *J. Inst. Metals* **82**, May 1954 456-462

Furnace Spectrum of Plutonium

197/I

United States. Little information is available on the plutonium spectrum and all results known were obtained with a high voltage spark technique. This method, however, gives little hope of separating the first and second spectra. A better method in this respect is the furnace method developed by King, although even here an appreciable ion spectrum is found at high temperatures. The author describes a modified furnace method which he employed for a study of the plutonium spectrum in the wavelength range from 3476 to 8400 Å. Furnace temperatures were 2600 to 2400°C., 2400 to 2200°C., 2200 to 2000°C. Wavelength and measured intensities are given in a table but between 6900 and 8400 Å no lines have been observed. The furnace used consisted essentially of a ¼-inch tantalum lined tube inserted into, and projecting from, a tubular graphite heater element situated in a vacuum chamber. 1000 amp and 11 volt heated the graphite element to 2250°C. After loading the plutonium in the form of a hydroxide slurried in water into the tantalum tube which was carried out in a ventilated gloved box and after adding caesium nitrate crystals to suppress ion spectra the tube was placed into the furnace and the furnace was evacuated and heated for a preliminary period to 800–1000°C. in order to convert the plutonium hydroxide to oxide. Subsequently the temperature was raised to the desired value and the measurements were taken.

Sommaire : Le spèctre du plutonium est déterminé par la méthode de King employant un four à vide modifié.

Article by J. G. Conway *J. Opt. Soc. Amer.* **44**, April 1954 276-278

Arc Melting Kroll Zirconium Sponge

198/I

See Abstract No. : 139/III

An Improved Method of, and Apparatus for, Determining Gases Contained in Metals

199/I

See Abstract No. : 134/III

Diffusion of Hydrogen in Mild Steel

200/I

Belgium. From experiments on the rate of removal of hydrogen from steel, it has been suggested that the hydrogen exists in 2 forms, with different effects upon the physical properties. This hypothesis is not confirmed by new experiments which show that there is reasonable agreement with expectation based upon the laws of normal diffusion. The equipment included a high-frequency induction furnace, immediate pumping of evolved gas by a high-capacity mercury diffusion pump, and collection of the gas into a calibrated volume in which the pressure could be determined by a thermoelectric gauge or by a McLeod gauge. Samples could be introduced and removed without breaking the vacuum. In the first experiment deposited weld metal analysing C 0.08, Si 0.40, and Mn 0.75% was heated to and maintained at 650°. It was estimated that the sample did not reach 500° during the first minute. The hydrogen extracted during the first minute was 0.33 ml./100 g. After 3 minutes the amount was 3.65 ml./100 g. After 5, 9, 12, 15, and 20 minutes the hydrogen extracted was 5.89, 7.41, 7.78, 7.96, and 8.22 ml./100 g., respectively. With this apparatus values obtained for the diffusion coefficient were $5.6–16 \times 10^{-3}$ sq. cm./min.

(*Chemical Abstracts*)

Sommaire : Une étude sur la mécanisme de la diffusion d'hydrogène dans les aciers doux chauffés.

A. Demarez A. G. Hock and F. A. Meunier *Acta Met.* **2**, 1954 214-223

Vacuum-Fusion Analysis of Molybdenum

201/I

See Abstract No. : 136/III

Thermionic Sintered Cathodes Made of Nickel and Alkaline Earth

202/I

See Abstract No. : 59/IV

Results of Experiments on the Behaviour of Nickel for Cathodes, Heated under Vacuum

203/I

See Abstract No. : 143/I

The Adsorption of Gases on Nickel Oxide

204/I

See Abstract No. : 181/I

II — Vacuum Apparatus and Auxiliaries — II

Abstract No. and References

20 — PUMPS — 20

Ionic Pump — 92/II

United States. An ion pump is described which consists of a 1-inch diameter molybdenum ring acting as the anode and held by a glass insulator between two thin carbon discs $\frac{3}{4}$ inch apart. An a.c. or d.c. voltage in the range of 1,000 to 10,000 V is applied between the ring and the discs, and an axial magnetic field of about 10,000 gauss is provided by an external permanent magnet. In operation the electrons produced in the discharge oscillate through the ring and ionise the gas molecules. These ions are attracted to, and caught by, the negative discs, at the same time producing secondary electrons which support the discharge. The pump has operated at pressures between 0.1 and 5×10^{-7} mm. Hg, and has absorbed up to 70 c.c. of air at N.T.P. without becoming saturated.

Sommaire : Des détails sont donnès sur un type de pompe ionique travaillant sur le principe de l'absorption.

Note by A. M. Gurewitsch & W. F. Westendorp *Rev. Sci. Instrum.* **25**, April 1954 389-390

The Physical Processes in Diffusion and Vapour Jet Pumps — 93/II

See Abstract No. : 139/I

Improvements i.o.r.t. High Vacuum Diffusion Pumps — 94/II

Liechtenstein. In high speed diffusion pumps with large diameters the distance from the top stage nozzle to the cold wall is necessarily great, with the result that the vapour density near the wall becomes too low to develop maximum pumping speed at high fine pressures. Also, since the jet must be able to expand downwards towards the cold wall, the height between stages becomes excessive. The patent describes a construction using two jet systems working in parallel which is claimed to overcome both difficulties. A cylindrical cooled partition is introduced inside the main cooled wall, and is about half its diameter. Inside this partition is a jet system of conventional pattern but having the reduced height associated with its smaller diameter. The second jet system is a hollow cylindrical structure of the same height completely surrounding the partition and symmetrical about its axis. The jet nozzle for each stage is here in the form of an annular ring which divides the vapour stream and directs it downwards in two separate jets, one expanding inwards to condense on the partition and the other outwards on to the main cooled wall. The pumped gas from both jet systems is led out through ducts to a common outlet port.

Sommaire : Description d'une pompe à diffusion équipée de deux systèmes d'injecteurs travaillant en parallèle; cette pompe est dite avoir une vitesse de pompage améliorée.

A. Vogt *Brit. Pat.* 709, 119

Electronic Controlled Pumping System — 95/II

United States. An automatic control system for the operation of a Toepler pump is described. A U-tube mercury switch has two electrical contacts, one at the bottom of the U-tube and the other at the top of one arm. The latter is connected by a pipe to the reservoir of the Toepler pump so that when the reservoir is under vacuum the mercury in that arm of the U-tube will rise until the two electrical contacts are shorted. Two contacts belonging to a second electrical circuit are placed on the body of the pump in positions, so that the circuit is closed when the mercury in the pump has risen high enough for the gases pumped to have entered a space fitted with a spherical non-return valve to prevent back diffusion. The reservoir of the pump is connected to a vacuum pump or to atmosphere, both lines being controlled by a magnetic valve, only one of which is open at a time. Details of the control circuit are given. It essentially consists of a conventional power supply which supplies a circuit consisting of three valves. A 6V6 valve has in its anode circuit a relay winding which, in the de-energised position, energises the valve controlling the vacuum pump line and, in the energised position, energises the valve controlling the line to atmosphere. The other two valves are of the 6SJ7 type and operate in such a manner that one becomes conducting when the U-tube circuit is closed and the other when the pump body circuit is closed. A condenser is arranged in a Y-network connected to the cathodes of the two 6SJ7 valves for the purpose of delaying the operation of one of them until the other is actuated and changes the state of operation of the relay in the 6V6 valve. A re-set switch is incorporated in the circuit which is depressed to energise the valve controlling the vacuum pump line thus starting the cycle of operation.

Sommaire : Un donne des détails sur un circuit electronique pourant être employé pour le controle et l'opération automatique d'une pompe Toepler.

D. S. Schover & Office Sci. Res. & Development, U.S.A. *U.S. Pat.* 2,682,364

Air Pump for Food Containers — 96/II

United States. A description is given of a hand-operated pump, integral with the lid of a food container, which can be used to reduce or increase the pressure within the container. The lid is screwed onto the threaded rim of the container to make an air-tight joint and the air is sucked out of the container into the cylinder of the pump through a spring-loaded ball valve which is screwed through the common part of the lid between the pump cylinder and the container. The piston of the pump works against a compression spring within the cylinder, so that little strength is required to operate the pump. The piston is also pierced by a tapped hole into which a second spring-loaded valve is screwed. The action of the two valves can be reversed so that the pump can be used as a compressor. A third valve, fitted in the lid of the container away from the pump cylinder, facilitates admission of atmospheric air.

Sommaire : Un petit équipement de pompage a été devéloppé pour la mise sous pression et le vidage de differents types de boites de conserve.

V. B. Lazerus *U.S. Pat.* 2,669,176

II — Vacuum Apparatus and Auxiliaries — II
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21 — GAUGES — 21

Improvements in Manostat 97/II

See Abstract No.: 128/II

Pressure Gauge for the Cm Range 98/II

United States. A modification which simplifies working is proposed for a gauge described by Alpert *et al.* previously (see Vol. I Abstract No. 133/II) which facilitated pressure measurements without the danger of contamination of the system by the manometric fluid. In the new design the flexible diaphragm is replaced by a metal bellows in a glass tube. The latter, closed at one end, is sealed at the other to a glass stem communicating with the system. The bellows carries pins at the closed end engaging in slots of a stirrup which restrict the movement of the bellows, the stirrup carrying at its extreme end a mirror on pivots. The latter is linked with the bellows and its tilt is read by a lamp and scale. The space surrounding the bellows is connected to a fore vacuum pump and a manometer. When gas is admitted to the vacuum side the bellows extends and the tilt of the mirror is recorded. The pressures are then equalised by allowing air to enter the measuring system until the mirror returns to its previous position. The pressure is then read on the manometer. At a distance of the scale of 1 metre, deflection is about 3 mm. per mm. Hg pressure difference.

Letter by J. B. Johnson & N. T. Williams *Rev. Sci. Instrum.* **25**, April 1954 393-394

Sommaire: Description d'une jauge de pression d'Alpert pour les mesures dans la région du cm. Hg, employant un soufflet en métal dans un tube de verre, au lieu d'un diaphragme flexible.

A Thermistor McLeod Gauge for a Pressure Range 1–10^{-7} mm. of Mercury 99/II

United Kingdom. Details are given of the design and performance of a sensitive pressure gauge made by sealing a thermistor bead into the end of the closed capillary of the McLeod gauge. The gauge has the characteristics of a Pirani-type gauge and can be used directly in the range of 1–10^{-3} mm. Hg. Lower pressures can be measured by enlarging the capillary around the thermistor bead to form a small chamber into which the gas is admitted until it reaches a pressure within the aforementioned range. The actual pressure in the system can then be computed from the measurement of the thermistor resistance and the compression ratio. A thermistor bead of the type Stantel U2361/20 which has a resistance of about 1,500 ohm at 20°C was employed in the construction of the instrument. Electrical connections were made by dipping the free ends of its platinum leads into small mercury cups. The matching thermistor was of the F2311/300 type which is available already enclosed in a glass envelope. Details of the electrical circuit are given. Calibration was carried out with an additional McLeod gauge. The volume of the thermistor chamber could be determined by compressing air of a given low pressure into it. As the volume of the thermistor gauge bulb was known the compression ratio could be calculated.

Article by R. S. Bradley *J. Sci. Instrum.* **31**, April 1954 129-130

Sommaire: Description détaillée d'une gauge de McLeod ayant une thermistance perle scellée à l'extrémité du tube capillaire de mesure.

A Quartz-Coated Wire Pirani Gauge 100/II

United Kingdom It would be advantageous to run Pirani gauges at high temperatures. In practice this is not feasible because tungsten and platinum, the two filament materials normally employed, show increased oxidation rate and random changes of accommodation coefficient respectively at temperature levels above 600°K. The author reports on the performance of a Pirani gauge, fitted with a quartz-coated wire, at high temperatures. The wire was 10 cm. long 0.001 inch diameter and the quartz coating was of a thickness of 0.002 inch. To facilitate electrical contact the end of the wire was acid-treated. The gauge was run at various temperatures between 600 and 1200°K. Details of the electrical circuit are given. The calibration showed great stability when the gauge was operated in air at pressures up to 1 mm. Hg for periods up to 30 days. Running the gauge at pressures in the mm. range for periods up to 120 days at 900°K, errors in gauge reading up to 12% were obtained. Generally, the experiments showed that Pirani gauges fitted with such wire, operating for periods not exceeding 100 hours at a time and employed for measurements in the pressure range up to 5 mm. Hg had an accuracy of ± 2%, when the temperature of the wire was 900–1,000°K. At high pressures and longer continuous operational periods the accuracy may be ± 5%. The zero drift is appreciably better than that of pure tungsten in the same temperature range. The new technique has the disadvantage that the wires employed must be straight and their effective length cannot be increased by helical construction.

Note by J. H. Leck *J. Sci. Instrum.* **31**, June 1954 226-227

Sommaire: On a étudié les avantages apportés par l'emploi d'un fil de platine recouvert de quartz, dans les gauges de Pirani, ce qui facilité l'opération de la gauge, à haute température.

Remarks on a Thermistor Pirani-Type Vacuum Gauge 101/II

Germany. One can use thermistors of small heat capacity to build vacuum gauges operating on the Pirani principle for a pressure range from 10^{1} to 10^{-3} mm. Hg. The difficulties which may arise from the use of thermistors are the strong dependence of the pressure measurement on room temperature, and the resistance tolerances to which thermistors are manufactured. It is shown how the room temperature effects can be satisfactorily reduced by the use of suitable compensating elements. As a result a vacuum gauge with satisfactory opera-

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tional characteristics is obtained. The thermostat elements considered are the so-called midget type N.T.C. resistance elements manufactured by Philips and 1–B and 1–D type thermistors produced by Westinghouse. They consist of globules of $\frac{1}{4}$ mm³ mounted on 5 mm. long and 50 μ thick platinum alloy electrodes. Their resistance is of the order of 1.5 kΩ at 20°C. In the proposed gauge two thermistors are mounted in a Wheatstone bridge network. One of them is exposed to the vacuum system and the other is at atmospheric pressure. The bridge voltage is stabilised by a 90 V stabilising tube.

Sommaire : Description detaillée d'un manomètre Pirani opérant avec des thermistances.

Article by H. Gruber *Glas- & Hochvakuum Tech.* **2**, Feb. 1954 302-306

Improvements i.o.r.t. Ionisation Gauges

102/II

United Kingdom. In the conventional type of ionisation gauge the electrons emitted from the filament ionise the gas, the pressure of which is measured, on their way to the anode. The ions formed are attracted by the ion collector and in the pressure range of 10^{-4} to 10^{-6} mm. Hg. this ion flow is proportional to the number of electrons passing from the filament to the anode. It has been found that in pressure ranges below 10^{-6} mm. Hg this proportionality no longer exists and consequently the gauge indication is no longer accurate. This deficiency of the gauge results from the fact that at these pressures the anode emits soft X–rays under the impact of the electron stream. X-rays reaching the ion collector will cause the latter to emit electrons. Each electron removed from the ion collector results in an apparent gain of the positive charge collected. In order to overcome this phenomenon it is proposed to surround the collector with a shield which is held at a negative potential with respect to the ion collector. This shield has a slot large enough not to obstruct the path of the ions formed in the discharge. In a particular application of this new design the collector is maintained at –2 V, the anode at +100 V and the shield at –40 V. Any electrons evolved from the ion collector due to X-rays originating in the anode are returned to the collector by the negative shield. This arrangement has been found to suppress 99.8% of the electrons emitted by X-rays from the ion collector.

Sommaire : Une gauge à ionisation a été spécialement modifiée de sorte qui les rayons-X mou émis dans la région des basses pressions sont supprimés.

G. H. Metson & H. M. Postmaster General *Brit. Pat.* 709,266

An Ionisation Manometer and Control Unit for Extremely Low Pressures

103/II

Canada. The lower operational limit of conventional ionisation gauge lies in the pressure range of 10^{-6} mm. Hg and below. The phenomena which cause inaccurate indication of the instrument in that pressure region are those described in Abstract No. 102/II. A method of overcoming the difficulties, alternative to the method employed previously, is that of reducing the surface of the collector. An example of the application of the latter method is given in the present paper. The principle of the new design is that of the Bayard-Alpert type ionisation manometer. The filament, consisting of a twin spiral of 0.01 inch diameter tungsten wire, is situated outside the electron grid which is wound in the form of a cylinder (cage) of 1 inch diameter and 1.5 inch length from 0.005 inch diameter molybdenum wire (10 T.P.I., on four molybdenum supports). The ion collector is a 0.003 inch diameter straight tungsten wire extending into, and along the axis of, the grid cage by 1.4 inch. The whole assembly is enclosed in a spherical Pyrex bulb to facilitate outgassing of the electrodes at high temperature. The operating characteristics of the new design are given in graphs. The sensitivity of the ion collector is effectively constant at potentials less than –25 V. At higher values the curves shows an upward slope indicating that ion trapping is not complete. Closing the ends of the grid cylinder improved ion trapping efficiency. A negatively charged shield grid surrounding the assembly and placed between the assembly and the walls of the envelope increased the path length of the electrons and thus improved the sensitivity of the instrument. The presence of this grid stopped the well-known pumping action of the gauge. However the shield was difficult to outgas and therefore was not incorporated in the final design. Details of the electrical circuit are given. The control unit is direct reading in pressure to 10^{-10} mm. Hg. The gauge was found to suppress photo-emission sufficiently well to facilitate operation of the gauge down to a pressure of 10^{-14} mm. Hg. The upper limit of the gauge is governed by space charge phenomena which begin to become effective at a potential of 150 V between grid and collector.

Sommaire : Description d'une gauge à ionisation pour mesurer des pressions de l'ordre de 10^{-14} mm. Hg. Les méthodes pour augmenter la sensibilité sont discuties.

Article by P. A. Redhead & L. R. McNarry *Canad. J. Phys.* **32**, April 1954 267-274

The Influence of Electrode Geometry on Cold-Cathode Vacuum Gauges

104/II

United Kingdom. Experiments have been made to examine the influence of the geometry of the electrodes on the performance of cold-cathode, or 'Penning', vacuum gauges. The arrangements reported include (*a*) disc cathodes and ring anodes, (*b*) ring cathodes and ring anodes, (*c*) disc cathodes and cylindrical anodes, and (*d*) disc cathodes and wire anodes. In addition gauges with multiple ring anodes and multiple cathodes have been examined. The bearing of the magnetic field on the performance of these instruments has been assessed. The broad conditions of stability are clear and it is shown that the pressure at which changes of mode take place can be controlled and, with some arrangements, eliminated at the expense of a small loss of sensitivity.

(*Authors*)

Sommaire : Des essais ont été faits en vue d'éxaminer l'influence de la forme géométrique des electrodes sur les performances de jauges à vide du type de Penning.

Article by G. K. T. Conn & H. N. Daglish *Vacuum* **4**, April 1954 136-146

A Sensitive Leak Detector Using Magnetron Ionisation Gauges

105/II

See Abstract No. : 130/II

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Abstract No. and References

22 — MEASURING PLANT (VACUUM) — 22

Economical Use of a Leak Detector Spectrometer 106/II

See Abstract No. : 141/I

Vacuum Requirement of the Beta-Ray Spectrometer 107/II

Belgium. The vacuum engineering problems involved in the design and operation of beta-ray spectrometers are discussed. In the particular case, the instrument chamber is made of aluminium and gate valves are provided to isolate the source and the counter respectively.

Sommaire : Les problèmes de technique du vide impliqués dans la fabrication et l'emploi des spectromètres à rayons beta sont discutés.

Letter by M. Legros *Vacuum* 4, April 1954 197-198

The Electron Microscope with Particular Reference to Its Use in Biological Research. I 108/II

United Kingdom. The maximum resolution and magnification obtainable in an electron microscope is 10 Å and x 200,000 respectively. This compares with a resolution of about 2,000 Å and a useful magnification of x 1,000 in the case of a good-quality light microscope, using visible green light. The elements of an electron microscope are similar to those of a standard cathode-ray tube but due to operational requirements there are important differences in design. Provision must be made for inserting specimens and photographic plates, and facilities must be provided for moving the specimen within the instrument chamber. In order to maintain the high vacuum required the chamber is pumped continuously. Variation of the illuminating intensity, focusing and adjusting of magnification are effected by changing the current passing through the solenoid-maintained magnets positioned co-axially with the electron beam and acting as the electron lenses. The image is first focused on a fluorescent screen but the latter is normally then replaced by a photographic plate in order to obtain the full benefit from the high resolving power of the instrument. Observations in the optical microscope are facilitated by contrast due to selective absorption of light by different parts of the specimen and the specimen support is normally glass which has negligible light absorption. But there are no substances which are transparent to electrons and the specimen to be examined in the electron microscope should be mounted on films or membranes thin enough to permit penetration by the electron beam. These films are supported by fine wire grids. When exposed to the electron beam, the specimen will scatter electrons and the specimen itself will appear as a dark area on the fluorescent screen. Should any appreciable absorption take place on the specimen, heat will be produced in that place and this may rupture the supporting film. The penetrating power of the electrons is governed by the velocity, expressed in terms of beam energy. The beam energies of commercially available electron microscopes vary between 40 and 100 electron kV. The penetrating power of the latter is about 0.5 μ. Various materials and methods for the preparation of specimen supports are discussed, including evaporated amorphous carbon films. All specimens must be desiccated and prepared in a very pure form. Freeze drying is sometimes used to fix the specimen but osmic acid fixation is less cumbersome and more frequently used. The preparation of various types of specimens are discussed in detail: (i) Large molecules, (ii) fibrous material (*e.g.* muscle and collagen), (iii) tissue and (iv) micro-organisms such as bacteria and virus and spermatozoa. Finally, the merits of shadow-casting are briefly reviewed.

Sommaire : Un article détaillé sur l'exécution et les applications de l'électromicroscope aux références particulières sur ses emplois dans les recherches biologiques.

Article by C. E. Challice *Lab. Practice* 3, June 1954 238-242

A Compact Console-Type Electron Microscope 109/II

United Kingdom. Details are given of an electron microscope, designed as a unit in desk form with the microscope tube in a horizontal position, which is to fill the gap between high-resolution electron miscroscopes and good-quality optical microscopes. It operates at 50 kV, facilitates magnifications from x 1,000 to x 10,000 and has the dimensions, 2 ft. 6 inch. square by 3 ft. 9 inch. high. The electron optical system consists of a biased electron gun and four electromagnetic lenses. The projector lens is of a special design which facilitates large magnifications to be obtained in a short stage length. The stage is provided with three positions and is designed to be moved in such a manner that the whole area of the 1/8 diameter grids can be searched. Changing of specimens introduces less than 1 c.c. of air which is removed by the pumps in a few seconds. The pumping plant consists of a 2-inch. diffusion pump coupled with a rotary backing pump. If the latter is of the 2-stage type, pumping times are halved. The plant incorporates a (diffusion pump) by-pass line, and two backing valves and a flap valve above the diffusion pump. All valve operations are controlled by a common control shaft allowing for four positions: (*a*) Normal operation, (*b*) temporary shut-down, (*c*) re-pumping and (*d*) final shut-down. Time required for re-pumping after a change of the films is about 10 minutes. The resolving power of the instrument is about 100 Å.

Sommaire : Ce papier decrit un microscope electronique plus petit, plus simple et meilleur marché que le modèle normal, développé pour des travaux routiniers en microscopie electronique.

Article by R. S. Page *J. Sci. Instrum.* 31, June 1954 200-205

Evacuating Photographic Materials for use in Electron Optical Apparatus 110/II

United States. The time spent on the evacuation of electron microscopes and diffraction cameras is very largely determined by gas evolution of the photographic films and plates used in these apparatus. Where large areas

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of photographic material are exposed, *i.e.* 100 to 1,000 cm²., the capacity of the pump and plant may have to be increased. The author investigated means of reducing gas evolution from photographic material after its insertion in the instrument. The material tested was plates (2 x 2 Eastman medium lantern slides with an anti-halation backing), and films (Eastman 5302 positive emulsion on a 35 mm. perforated acetate base). The tests were carried out in a R.C.A. E.M.T. electron microscope and also in a small specially built vacuum system. As the maximum operating pressure of kinetic electron optical systems is 0.5 micron Hg, the time taken to evacuate these materials, pre-treated by various methods, to this pressure were observed. Water vapour was found to be the major constituent of the gases and vapours evolved from the photographic material. It is important therefore to desiccate these materials effectively prior to use. This was effected in three ways: (*a*) By exposure to a desiccant, (*b*) by heat treatment and (*c*) by pre-pumping. (*a*) proved to be the least effective. The desiccating agent used was anhydrous calcium sulphate (Drierite). If the material was enclosed in a container such as a 35 mm. daylight-loading spool, desiccation was very slow. (*b*) is a delicate process because of the potentially deleterious effects of high temperature on the photographic emulsions. During a four-hour treatment the temperature should not exceed 120°F. (*c*) Is the most effective form of pre-treatment. The time required to transfer the material from the desiccating apparatus to the instrument is a critical factor as during exposure to atmosphere rapid resorption of water vapour takes place. Pre-pumping below 0.5 micron Hg is useless because the material treated in this manner will take up water vapour so rapidly that all benefits of the treatment are lost. Evacuation to 0.5 micron Hg will have its full effect only if exposure to atmosphere is restricted to a few minutes. Generally speaking plates were found to dry faster than film. Detailed results are given in graphs.

Sommaire : Afin de reduire le temp de pompage dans les appareils à optique électronique l'emulsion photographique doit être séchée auparavant, ceci, soit au moyer d'un produit desséchant, soit par pompage ou élévation de température.

Article by J. H. Reisner *Rev. Sci. Instrum.* 25, May 1954 477-479

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Electron Microscopy of Photographic Grains. Specimen Preparation Techniques and Applications 111/II

United States. The effects of exposure and development on silver halide grains in the electron microscope have been investigated before but little is known yet on the mechanism of latent image formation. In the present paper several specimen preparation techniques are described which were developed for the investigation of a number of negative, high-contrast, double-coated, black and white emulsions with a mean grain diameter of about 0.75 μ, such as a fast bromoiodide-ammonia emulsion of relatively high ratio of surface to internal sensitivity. In all cases the emulsion was a matrix of gelatine supported by a cellulose ester film base. In order to facilitate the examination of the grains, the inter-grain gelatine was removed by enzymatic hydrolysis as a first step. Details of the technique applied are given. The end product was found to consist of grains surrounded by a highly transparent thin silver-gelatinate film, termed by the authors' 'combined envelope'. A methodic study of the structure and properties of the combined envelope led to the development of methods whereby the combined envelope is utilised to retain the latent image sufficiently enlarged to be readily resolvable in the electron microscope.

Sommaire : Des matériaux photographiques ont été étudiés au moyen du microscope électronique.

Article by F. A. Hamm & J. J. Comer *J. Appl. Phys.* 24, Dec. 1953 1495-1513

An Improved Replica Technique for Electron Microscopy of Paint Films 112/II

See Abstract No. : 186/I

A Simple Method of Preparing Silica Replicas. Advantages Offered by the Cathodic Etching of Specimens 113/II

France. The majority of the methods known for the preparation of silica replicas of surfaces of a specimen requires the application of a double-replica technique. The authors give details of a single-step technique which has been successful. The specimen is placed in an evaporation chamber, 13 cm. away from a tantalum crucible. The amount of silica evaporated is calculated to give a deposit of about 100 Å thickness. Evaporation has to be rapid to facilitate easy removal of the silica film from the specimen. After completion of the evaporation a 1%– collodion solution is applied to the silica film and after removal of the solvent the compound film is withdrawn from the specimen by means of cellotape. The film is cut into small squares, which are immersed in acetone to dissolve the collodion. Then, the silica film squares are dropped into a beaker filled with distilled water, picked up w.th the help of a grid, dried and shadowed. The method can also be applied for the production of silica films which are to serve as specimen supports. Such films, deposited on glass, are completely structureless. The authors conclude with remarks on the uses of cathodic sputtering as a means of etching the specimen surface in order to facilitate easy removal of the replica film.

Sommaire : Description d'une méthode facilitant l'obtention directe de replicas de silice de surface d'échantillon. La gravure cathodique est mentionnée.

Note by C. Fert & R. Dargent *C. R. Acad. Sci. Paris* 238, 8.2.1954 662-664

26 — MATERIALS — 15

Ionisation and Excitation of Mercury Vapour by Positive Ion Impact 114/II

See Abstract No. : 156/I

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Abstract No. and References

Fused Vacuum-Tight, Metal-to-Ceramic, Ceramic-to-Glass, Metal-to-Glass and Metal-to-Mica Sealing by Powdered Glass Techniques

115/II

See Abstract No. : 62/IV

Experiments in the Design of Ceramic Electron Tubes

116/II

United States. Experiments have been carried out on the production of vacuum-tight ceramic-to-metal seals with the object of developing a ceramic diode. The uses of the following two commercially available glazed ceramics were studied: (*a*) A zirconium-silicate base known as Zi4 (Coots Porcelain Co.) and (*b*) a magnesium-silicate base AlSiMag 243 (American Lava Co.). A search for metals with an expansion coefficient similar to that of the two ceramics considered showed that Kovar and, to a greater extent, molybdenum were suitable for use with (*a*), and a nickel-iron alloy matched (*b*) over a wide temperature range. Among the sealing methods considered were: (i) The Bondley technique, in which high vacuum and r.f. heating are used, employing silver as a brazing agent, (ii) the technique, in which a layer of molybdenum-iron silicates is fired on to the ceramic, a nickel powder being then sintered on the surface to provide a base for brazing agents such as silver, and (iii) a new technique, where the ceramic and metal parts are drawn together in a vacuum at high temperatures. The authors actually employed the Bondley technique, a detailed description of which is given: Both ceramics were used in the experiments. The greater part of the seals made by this method were circular. The end of the ceramic cylinder was coated with ZrH_2 and a ring of silver wire placed around the cylinder near the end to be sealed. The assembly was placed on a metal disc and lowered into a carbon crucible which in turn was suspended in a glass envelope which could be evacuated. A glass 'umbrella' was used to protect the walls of the vessel from the deposition of silver during the sealing process. After evacuation of the receptacle to about 1 micron Hg, r.f. heating was applied and the temperature raised slowly to allow for the removal of the evolved gases by the pumps. Completion of the sealing process was indicated by the formation of a thin film of silver on the umbrella. The temperature was then reduced slowly and the pressure brought up to atmosphere. An alternative procedure, operating in an atmosphere of hydrogen, nitrogen or neon, using a cylindrical quartz oven and ordinary oxygen-gas torches as heaters, proved to be quicker, but the quality of the seal obtained in the vacuum procedure was higher. The best seals were those between ceramic (*b*) and the nickel-iron alloy mentioned. These seals, therefore, were chosen for the production of the experimental ceramic diodes. The vacuum properties of these tubes were found to be satisfactory. A pressure of 10^{-7} mm. Hg could be maintained in the tubes without trouble resulting from leakage. The main difficulties encountered in the construction of the diodes originated from the gettering techniques employed and the occurrence of electrical leakage due to condensed Zr-Ag vapour. Tubes manufactured in the manner described are of light weight, have a good high-temperature strength and performance characteristics which makes them suitable for application as high-power rectifiers or transmitting tubes.

Sommaire : Des essais out été faits sur la production de joints étanches au vide céramique-métal dans le but de développer une diode en céramique.

Article by F. Brand H. Jacobs & C. LoCascio *Ceramic Age* **63**, May 1954 18-23

Use of Toggle Valves and Teflon in Hydrocarbon Analysis

117/II

United States. Apparatus is described which is employed for the sampling of hydrocarbon gases to be analysed in the mass spectrometer. Details are given of the precautions taken to reduce any errors in preparing the samples. One persistent cause of contamination of the samples was traced to the sorption properties of stopcock grease which caused hydrocarbons sorbed during previous tests, to desorb during subsequent tests and contaminate fresh samples. It was decided therefore to remove all stopcocks in the system and to employ instead small needle toggle valves fitted with Teflon seats. Although the new valve design still incorporates a small rubber O-ring seal, no hydrocarbon cross contamination occurred since the introduction of the valves. The use of valves instead of stopcocks required the provision of glass-to-metal connections. A Teflon packing gland connection was designed for the purpose, which was more convenient than the soldering techniques normally employed. Details of the valves and the packing gland connections are given. Valves designed as described have been in use for nearly two years without showing leakage. The occasional leak was eliminated by heating the valve in the closed position and rotating the stem in order to reseal the valve seat.

Sommaire : On donné des détails sur une petite vanne à aiguille avec siège de Teflon, employées à la place des robinets d'un appareil de manipulation des gaz échantillions d'hydrocarbons liquides.

Note by R. A. Brown & D. J. Skahan *Analyt. Chem.* **26**, April 1954 788-789

Physical Instability and Thermal Shock in Red Cells

118/II

United Kingdom. The use of media containing glycerol has made possible the preservation of many types of living cells in the frozen state. The presence of glycerol prevents the formation of highly concentrated salt solutions during freezing which disperse lipids and lipoprotein from the cell membrane leading eventually to the dissolution of the cell. Glycerol does not completely prevent an increase of salt concentration and evidence suggests that the long-term exposure of relatively high concentrations of salts in glycerol media gives rise to slow deterioration of the frozen cells. As a study of the deterioration of cells at low temperatures takes a long time, higher temperatures were used in the experiments and the cells were kept in media which were maintained unsaturated with respect to the components of their membranes. It was found that the rate of haemolysis fell between 40°C and 5°C and rose below 4°C, suggesting some change at this temperature. Loss of lipids by simple diffusion appears to cause no great harm to red cells. Loss of phospholid was, however, found to render these cells subject to rapid lysis on cooling below 20°C. This appears to be a manifestation of the same phenomenon

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that causes thermal shock in mammalian spermatozoa. Treatment with lecithin can reduce the effect of both. The effect is attributed to some physical change in the cell membrane at low temperature and a tentative explanation of the probable mechanism is given.

Sommaire : Les effects de milieux contenant du glycérol sur certaines cellules vivantes préservées à l'état gelé, sont discutés.

Article by J. E. Lovelock *Nature* **173**, 10.4.1954 659-661

28 — APPARATUS COMPONENTS — 28

Techniques Useful in Evacuating and Pressurising Metal Chambers 119/II

See Abstract No. : 163/I

Seal-Off Valve for Vacuum Systems 120/II

United States. Details are given of a small seal-off valve which may be employed with advantage where the use of a conventional valve is undesirable because of its bulkiness, and pinched-off metal tubing is not practicable because a new piece of tubing has to be soldered on to the system each time it is evacuated. The main parts of the new valve are a handle and stem, body and seat, and a removable bonnet enclosing the seat assembly and adjacent end of the stem. The moveable seat has six pumping ports, through which the system is evacuated. On completion of the pumping the stem, fitted with pins engaging in two of the pumping ports, is used to close the valve. The bonnet is then removed, leaving only the seat assembly in position on the pipe. To open the valve, the procedure is reversed.

Sommaire : Description d'une vanne de fermeture dont la poignée et l'axe peuvent etre facilement enlevés laissant seulement le système de fermeture sur l'appareil vidé.

Report by Anon. *NBS Summary Tech. Rep. No.* 1858 May 1954

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Use of Toggle Valves and Teflon in Hydrocarbon Analysis 121/II

See Abstract No. : 117/II

A Three-Way Vacuum Valve 122/II

United States. A three-way vacuum valve employing sliding O–ring seals is described. The valve has a hollow, cylindrical body with two peripheral O–rings set inside it, to divide the body into three equal sections. The O–rings act as seals against a central piston which is slightly longer than the distance between the rings. Positive seating is provided at each end of the piston stroke. The vacuum connections are on either side of, and in between, the O–rings in the body. The shaft and the body are extended beyond the distance between the seals. A pin in the shaft engages in a helical slot in the cylinder so that rotation of the shaft moves the piston in the axial direction.

Sommaire : Description d'une vanne à vide à trois passages employant des joints mobiles du type 'O–ring'.

Note by F. S. Stein *Rev. Sci. Instrum.* **25**, May 1954 515-516

A Magnetic Mercury Cut-Off 123/II

United Kingdom. A mercury cut-off is described, in which a glass cylinder closed at both ends, containing an iron bar, floats on mercury in a vertical tube and can be drawn beneath the surface by an electromagnet. The mercury displaced by the submerged glass cylinder rises in the tube and seals off a side arm. The cut-off will seal efficiently against a 2 cm. Hg pressure difference.

Sommaire : Description d'un système de fermeture à mercure pouvant tenir une difference de pression de deux centimètres de mercure.

Note by F. W. Thompson *J. Sci. Instrum.* **31**, April 1954 145

Cold Traps for Vacuum Systems 124/II

United Kingdom. The U–tube type of cold trap, immersed in a cooling medium such as liquid air, commonly used in vacuum pumping systems to prevent vapours from diffusing back into the vessel pumped, is not suitable in plant operating at high pumping speeds. Traps included in such plant should offer minimum obstruction to the flow of the gases coupled with the maximum probability of condensing all vapour molecules reaching the trap. In addition, they should be easily cleaned and degassed, and capable of being cooled rapidly. The design of a trap, claimed to meet these requirements, is described. It essentially consists of baffles situated in the pumping line which are cooled by conduction along a metal rod, one end of which acts as the support of the baffles, the other, projecting outside the pipe, being immersed in a vessel holding the cooling medium. The baffle system consists of a series of semi-circular sheets arranged in such a manner that the gases passing through the pipe can no longer move along a straight path but are subjected to tortuous flow. The baffle assembly is fitted into a flanged section of pipe which gives a clearance of 1–2 mm. all round the baffles, the pipe section being connected directly into the pumping line. The internal walls of the pipe should be polished to minimise heat radiation to the baffles. By way of example the following dimensions are given: Diameter of the pipe section—

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Abstract No. and References

3 inch., thickness of the baffles—0.028–0.030 inch. A trap of these dimensions has been found to have a conductance for air of 48 litres per second and the pressures measured directly above the trap were less than 10^{-7} mm. Hg. The cooling medium, in that instance, was liquid air.

Sommaire : Un donne des détails sur un piège composé de baffles refroidés par conduction le long d'une tige de métal, les baffles étant disposés dans la tubulure réliant le système à la pompe à vide.

J. Pollard & Nat. Res. Dev. Corp. *Brit. Pat.* 709,819

Apparatus for Stirring a Reaction Mixture Through a Vapour-Tight Seal 125/II

United States. Agitation of a liquid in a totally enclosed reaction vessel can be effected by a magnetic stirrer or, where this is inadequate, by a mechanically driven stirrer, in which case a seal of some kind is required for the driving shaft. A known form of seal of this kind, frequently used where the reaction vessel is kept at a reduced pressure, consists of a spherical glass joint, one part of which is stationary and the other rotates about the axis of the stirrer shaft, to which it is attached. The author describes an improved design of the latter type of seal which ensures symmetrical wearing of the seal. In the new design the stirring rod is sealed to the joint as before, or is attached by means of a rubber sleeve to allow for adjustment or replacement. The rod is free to turn about its own axis but is held in a counter-balanced arm of a drive which revolves it about a centre of rotation lying on an axis of the spherical joint. As a result, inside the flask, the stirring rod in action describes a cone. The zone of the reaction vessel covered by the action of the rod depends on the length of the driving arm and its height above the seal. Suitable speeds are 50–300 r.p.m. Units of this kind have operated successfully at pressures of 0.05 mm. Hg in the reaction vessel for long periods. The seals should be lubricated with a suitable lubricant.

Sommaire : Description d'un nouvel agitateur employé particulièrement pour aider des réactions se faisant dans des systèmes complètement fermés.

Note by J. R. Skidmor *Analyt. Chem.* **26**, April 1954 789-790

Experiments in the Design of Ceramic Electron Tubes 126/II

See Abstract No. : 116/II

Fused Vacuum-Tight, Metal-to-Ceramic, Ceramic-to-Glass, Metal-to-Glass and Metal-to-Mica Sealing by Powdered Glass Techniques 127/II

See Abstract No. : 62/IV

29 — MISCELLANEOUS APPARATUS — 29

Improvements in Manostat 128/II

United States. A manostat is described which is capable of memorising, after a rest period, a pressure to which it was set in an operation, *i.e.* if an operation in a system was carried out at a set pressure and discontinued, the manostat will return the system to the previous pressure when operations have been started again. The instrument consists essentially of a cylindrical housing containing a bell-shaped float with its open end dipping in mercury. The upper end of the float carries sealing discs of rubber-like material which, if the float rises high enough, will seal off a nozzle projecting downwards. The nozzle communicates *via* channels in the upper portion of the instrument housing with a vacuum pump on one side and the system to be pumped on the other. On the other hand, if the float sinks down low enough, the sealing discs at its upper end will close off a nozzle projecting upwards from a stationary tube of smaller diameter than the float and situated inside the float. This tube is connected to the system to be pumped *via* a valve. During initial pumping of the system the gases pumped from the system pass through the top portion of the instrument housing and reach the pump *via* the nozzle projecting downwards. When the pressure has been reached at which the manostat is to be set the position of the upper nozzle is adjusted in such a manner that the sealing disc of the float and the upper nozzle come in contact. The aforementioned valve connecting the space underneath the bell is then closed and thus traps an amount of gas in that space which acts as the memorising control gas. The instrument is then permanently set. When the system is shut down and opened to atmospheric pressure, the bell is forced down and the sealing disc, moving away from the upper nozzle, is pressed against the lower nozzle thus trapping the control gas. On restarting pumping, communication between the system and the vacuum pump will remain open until, through the action of the control gas, the upper nozzle is closed at the set pressure value. Various embodiments of the working principle are described.

Sommaire : Un manostat, capable de se rappeler une pression réduite particulière a été développé ; le manostat réadjuste le système remis en marche, à la pression, à laquelle il a été préalablement réglé.

Emil Greiner Co. *Brit. Pat.* 706,777

Economical Use of a Leak Detector Spectrometer 129/II

See Abstract No. : 141/I

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

A Sensitive Leak Detector, Using Magnetron Ionisation Gauges

130/II

United Kingdom. The experimental development of a leak detector using differential operation of two magnetron ionisation gauges is described. The gauges are connected in an a.c. bridge circuit and modulation is introduced by superimposing a small a.c. magnetic field on the d.c. field. The detector is particularly adapted for use with clean and outgassable vessels such as thermionic valves and cathode-ray tubes. Under these conditions, leaks of less than 10^{-7} lusec could be detected and the instrument is therefore as sensitive as commercial mass spectrometer leak detectors, while being much cheaper.

(*Authors*)

Sommaire : Description d'un detecteur de fuites experimental fonctionnant par opèration différentielle de deux jauges à ionisation à magnétron.

Article by A. H. Beck & G. King *Vacuum* 4, April 1954 147-158

The Design and Construction of Simple Linde Hydrogen and Helium Liquefiers

131/II

United Kingdom. High vacuum and low temperature techniques are closely related, but the latter are not widely used. In particular, the general methods for calculating the efficiencies of heat exchangers used with small liquefiers seem to be the usual stumbling block. These have been summarised. The advantages of a low resistance path for the gas are discussed, and the construction of a simple variation of the Hampson exchanger is given. By using small Linde hydrogen and helium liquefiers, experiments in the corresponding temperature ranges can be carried out on a limited scale without great expense or highly skilled workshop effort.

(*Author*)

Sommaire : Discussion des aspects essentiels et du fonctionnement pratique et théorique de petits liquéfacteurs d'hydrogène et d'helium.

Article by D. H. Parkinson *Vacuum* 4, April 1954 159-167

The Continuous-Diffusion Chamber

132/II

See Abstract No. : 168/I

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III — Vacuum Processing Techniques — III

Abstract No. and References

30 — EVAPORATION — 30

Notes on Solution of Problems in Odd Job Vapour Coating 102/III

See Abstract No. : 151/I

Evaporated Metal Films 103/III

See Abstract No. : 149/I

How to Finish Metal Products by Vacuum Metallising 104/III

See Abstract No. : 150/I

Aluminising TV Tubes 105/III

United States. Three types of aluminising plant employed by the General Electric Company, N.Y., for the production of 27-inch. all-glass TV tubes is briefly described. The first two, employed for the aluminising of black-and-white tubes, make use of self-contained buggies which are fitted with an oil diffusion pump coupled with a mechanical pump, an electric power supply to pre-heat the filament source and flash the aluminium, and a suitable fixture to support the tube and seal its funnel against atmosphere. The only manual operations required are the insertion of the filament source and aluminium charge assembly, and placing the tube in position. Processing is fully automatic and covers the full cycle from the rough-pumping stage to the admission of atmosphere after completion of the aluminising. In the first method individual buggies are used; in the second 24 buggies are mounted on a continuously revolving turntable known as a merry-go-round. The latter type of plant facilitates the aluminising of 100 TV tubes per hour. The third method is concerned with the production of colour TV tubes. It facilitates the aluminising of the phosphor plates as separate components. Three plates are simultaneously aluminised in a three-cornered bell jar. The presence of a conductive layer, such as is produced by aluminising on the back of the phosphor plate, ensures a uniform potential during the operation of the TV tube and thus assists in the maintenance of colour purity.

Sommaire: Note sur les méthodes employées pour aluminiser les tubes de TV dans un grande firme de fabrication.

Note by Anon. *Electronics* **27**, June 1954 260, 262, 264, 266

Vacuum Evaporation of Aluminium 106/III

United States. The life of basket-shaped tungsten filaments, used for vacuum evaporation of aluminium, may be lengthened by inserting a replaceable tantalum lining, formed from 10-mil sheet, into the crucible. The tungsten wire is electrolytically cleaned, then painted or sprayed with high-temperature alundum cement, dried in air and finally baked at a temperature not exceeding 1,500°C in high vacuum. The tantalum insert has to be replaced after one or two evaporation runs but the tungsten basket can be used several times.

Sommaire: La durée de vie des filaments de tungstène du type 'panier', employés dans l'évaporation d'aluminium, peut être augmentée en insérant dans le creuset une feuille de tantale.

Letter by R. L. Kelly & P. F. Rice *Rev. Sci. Instrum.* **25**, April 1954 391-392

Improvements in Metal-Coated Recording Strips 107/III

See Abstract No. : 160/I

An Investigation on Aluminium Electrolytic Capacitors and the Development of a New Type of Aluminium Oxide Capacitor 108/III

See Abstract No. : 159/I

Method of Producing Graded Coatings 109/III

See Abstract No. : 171/I

On the Simultaneous Determination of the Optical Constants and of the Thickness of Thin Absorbing Films 110/III

France. A new graphical method is described which enables simultaneous determination to be made of optical constants and the thickness of a thin film of a metal. This makes use of the measurement at normal incidence of reflection coefficients, transmission and, in certain cases, the change of phase. The phase change measurement is necessary when very thin films or very high absorption coefficients are involved. It is shown that the properties of thin films can be explained by electromagnetic theory if it is postulated that the optical constants vary with the thickness. The theory is confirmed by observations made on thin films of silver and gold, produced both by cathodic sputtering and thermal evaporation. The values found differ somewhat from older values and

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

it is demonstrated that the density of a deposited film is not equal to that of the bulk of metal, a fact which explains earlier errors. For gold the thermal evaporated and sputtered films have similar constants which differ from that of the bulk metal. An apparatus is described for the simultaneous production of five thin films of different thickness.

(*Science Abstracts*)

Sommaire : Description d'un nouvelle méthode graphique qui permet de déterminer simultanément les constantes optiques et l'épaisseur de films minces de métal.

Article by D. Male *Ann. Phys. Paris* 9, Jan.-Feb. 1954 10-51

Multi-Layer Reflecting Films at Wavelengths Between 4,000 and 4,600 Å

See Abstract No. : 173/I

111/III

The Optical and Electrical Properties of Cadmium Sulphide Films

112/III

Japan. The optical and electrical properties of thin evaporated cadmium sulphide films were measured. The optical properties such as the absorption edge, the spectral sensitivity of their temperature dependence are well reproducible and can be regarded the same as for single crystals of cadmium sulphide. The electrical properties such as the conductivity, the photosensitivity, and their temperature variation are very sensitive to the conditions of evaporation and to after treatments of the layers. Dark- and photo-currents are both nonlinear when plotted against the applied voltage, which suggests granularity of the film and the existence of a potential barrier between the grains. Activation energies derived from plots of the conductivity *vs.* the inverse temperature show variations between 0.06 and 0.28ev depending on the thickness of the sample. Freshly prepared thick films show high conductivity, low activation energy, and large photo-response. On baking in air above 200° the room-temperature conductivity decreases and the activation energy greatly increases. This effect is believed to be due to the adsorption of oxygen on the surface because the original conductivity can be restored by baking the film *in vacuo* above 200°.

(*Chemical Abstracts*)

Sommaire: Les propriétés électriques et optiques de films minces de sulfure de cadmium ont été étudiées.

Article by G. Kuwabara *J. Phys. Soc. Japan* 9, 1954 97-102

Improvements i.o.r.t. the Production of Indicia on Solid Bases, Such as Cross-Wires

113/III

Leichtenstein. A method, employing vacuum evaporation techniques for the production of scale graduations or similar marks, *i.e.* fine lines, grooves or sharply defined bands on lenses or plates, is described. Previous methods, including engraving as one processing step, resulted in marks lacking definition, chiefly originating from faults inherent in the action of the engraving stylus. Any irregularity of outline produced by the cutting tool is eliminated in the present method, and clear-cut side faces of the mark, substantially vertical to the base, are obtained. To prepare a 'cross wire' for instance, the base is coated with a brittle, non-hygroscopic and easily soluble material such as a fluoride, in particular aluminium or sodium fluoride to a thickness of less than 10,000Å. For very fine marks the thickness should be appreciably smaller. The outline of the required mark is engraved in this coating and a second film of different material is deposited. Due to the action of the engraving tool the second coat is in direct contact with the base along the outline of the mark. The top coat should be either insoluble in a solvent which will dissolve the first coating or soluble only under different conditions. Rhodium, which is suggested as a suitable material, has a light absorption of 30–40 % at a film thickness of 100 Å. The first coating is then dissolved. As a result the second coating is removed in all areas, where it was supported by the former and breaks away cleanly from the strip of material in direct contact with the base, *i.e.* along the outline of the marking. If the second coating is partly transparent, interference phenomena give the indicia a 'colour' determined by the thickness of the coating. A suitable adaptation of the method facilitates the production of transparent grooves in an otherwise opaque coating and the preparation of bands of material which abut along clear-cut edges. Indicia of the band-type may be used for colour screens or diffraction gratings. The method is claimed to facilitate the production of striae of a width of 2/1,000 mm. or less with clearly defined edges.

Sommaire: Description d'une méthode employant la technique de l'évaporation sous vide pour la production de fines lignes, etc., sur les échelles gradués d'élements optiques.

A. Vogt *Brit. Pat.* 707.149

Improvements i.o.r.t. Electrodes for Photo-Electric Devices

See Abstract No. : 174/I

114/III

Tentative Explanation of the Mechanism of Conductivity in Thin Metal Films of Granular Structure

115/III

France. The deviation from Ohm's law, exhibited by thin metal films of granular structure, has been explained by assuming the film to consist of a mass of insulated particles which exchange electrons through a potential barrier by the tunnel effect. However this interpretation is satisfactory only if the inter-granular distances are smaller than 30 Å. The author has studied these phenomena experimentally and come to the conclusion that the film should be considered to consist of insulated atoms of the metal, oxides, diffused impurities from the support and adsorbed gas, *i.e.* to be a typical semi-conductor rich in impurities. This facilitates the application of the classical theory of contact barriers in metal semi-conductors. The experiments conducted on germanium support this hypothesis, indicating hysteresis characteristics typical of rectifier contacts.

Sommaire : On donné une tentative interprétation des propriétés électriques de films de métal à structure granulaire.

Note by N. Nifontoff *C. R. Acad. Sci. Paris* May 1954 1870-1872

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Abstract No. and References

Semi-Conducting Films of Antimony

116/III

United States. The electrical properties of thin films has been investigated frequently in the past and their characteristics have been attributed to various causes. In order to establish the mechanism of semi-conductivity in thin antimony films the author deposited more than 40 films, varying the operational conditions in such a manner that measurements could be carried out to establish, (*a*) the effect of temperature of deposition on the temperature coefficient of resistance, (*b*) the effect of temperature after deposition on resistance and (*c*) the effect of evaporation rate and film thickness on activation energy. Evaporation was carried out in a glass tube at 10^{-5} mm. Hg. The substrate was kept at the temperature of dry ice or liquid oxygen during deposition. The cooling was effected by blowing nitrogen gas which was previously cooled in a liquid oxygen bath over the substrate. The temperature control was obtained by adjusting the rate of the gas flow. Film thickness was determined by calculation. Evaporation rate was controlled by varying the supply to the filament. Investigating (*a*), it was found that the source—substrate distance influences the sign of the temperature coefficient of resistance. At 4 cm. distance and at liquid oxygen temperature of the substrate the sign was negative, but at room temperature the sign was positive. At 8 cm. distance the sign was negative for all substrate temperatures. Investigating (*b*), it was found that cooling the films below substrate temperature during deposition tended to produce reversible resistance changes, while heating the films above that temperature produced irreversible changes. The results obtained while investigating (*c*) are given in a table partly reproduced below.

Calculated Activation Energies for Films of Various Thicknesses and Evaporation Rates

Film Thickness	*Evaporation Rate*	*Activation Energies*	
		Low Temperature	*High Temperature*
250 Å	0.8 Å/sec.	0.07 ev	0.13 ev
370 Å	0.9 Å/sec.	0.07 ev	0.13 ev
400 Å	0.8 Å/sec.	0.06 ev	0.12 ev
800 Å	9.0 Å/sec.	0.08 ev	0.14 ev

An analysis of the results permits the following tentative explanation of the mechanism involved. The initial layers of the deposit are amorphous and the pattern of the deposit at this stage is irregular, *i.e.* interatomic distances vary. The energy required to produce a more ordered deposit and/or crystallisation is supplied by the impinging beam or by the substrate. The former is lost by impact and the latter is not available if the substrate is kept cold. Thus it can be assumed that there are lattice defects in all metal deposits, in particular in those effected on a cold substrate, and that these lattice defects are the cause of the high resistances observed. These defects further account for the behaviour of the resistance in relation to temperature.

Sommaire : Des films d'antimoine furent déposés à différentes températures. L'effet de la température pendant et après l'évaporation sur la resistance des films, a été étudié.

Article by J. Cohen *J. Appl. Phys.* **25**, June 1954 798-801

Light Transmissive Electrically Conducting Optical Article

117/III

United States. The application to glass of an electrically conducting coating suitable for lenses, goggles, windshields, etc. which are used in freezing weather is described. The purpose of the coating is to prevent ice formation, resulting from the condensation of warm air, without an appreciable loss in light transmission. The deposition technique employed for the production of such electrically conducting coatings is essentially describedin British Patent 682,264, (see Vol. III Abstract No. 13/III). The present patent gives some additional information on the application of these techniques.

Sommaire: Une méthode de revêtement de pare-brise avec des films conducteurs d'électricté est discutée.

W. H. Colbert W. L. Morgan A. R. Weinrich & Libbey-Owens-Ford Glass Co. *U.S. Pat.* 2,676,117

Magnetic Behaviour of Thin Single-Crystal Nickel Films

118/III

See Abstract No. : 176/I

The Cause of Stress in Evacuated Metal Films

119/III

United States. The authors refer to previous work on stress in evaporated metal films published by Murbach and Wilman, (see Abstract No. 7/III), who proposed that the filmis stress-free at the recrystallisation temperature during deposition but that thermal expansion, occurring while the substrate cools down, introduces stress. The present authors report on experiments which indicate a different mechanism, *i.e.* the total stress present in a film is the sum of two components: (*a*) Resulting from differential expansion during cooling down of the substrate and (*b*) resulting from imperfections in the deposit, *e.g.* trapped gas molecules or irregular crystal structure. The stress due to thermal changes is found to be small compared with the 'intrinsic' stress. In the experiments, nickel films of 1,500 Å thickness were evaporated on to thin mica substrates at a pressure of 10^{-6} mm. Hg and at a rate of 60 Å/sec. Upon cooling to 25°C, the total stress was determined by measuring the deformation or curvature of the mica substrate, supported on knife edges. The value for (*a*) was found by measuring the

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deformation at the substrate temperature during deposition and again at 25°C. At 75°C deposition temperature (a) was 0.4×10^9 dyn/cm². The intrinsic stress was determined as a function of both substrate deposition temperature and annealing temperature. The results for four nickel-mica specimens, produced at different substrate temperatures, are shown in a graph demonstrating that annealing reduces intrinsic stress, reaching a minimum value at 25°C and rising again thereafter. If the concept of Murbach and Wilman applied, the film should be stable during annealing at lower temperatures. It is concluded that the intrinsic stress in a thin metal film can be related to the imperfections formed during deposition which undergo changes shortly after being trapped by succeeding layers of deposited metal. The density of the initial imperfections appears to depend on the surface mobility of the evaporated metal. Copper shows low intrinsic stress. Its surface mobility is known to be higher than that of nickel. A table, partly reproduced below, gives comparative data for nickel and copper.

Metal	*Ni*	*Ni*	*Cu*	*Cu*
Substrate deposit temp (°C)	75	125	25	75
Total stress (10^9 dyn. cm.$^{-2}$)	6.80	4.70	0.18	0.37
Diff. exp. stress (10^9 dyn. cm.$^{-2}$)	0.40	0.94	0.0	0.33
Intrinsic stress (10^9 dyn. cm.$^{-2}$)	6.40	3.76	0.18	0.04

Sommaire: Les causes de tension dans les films de métal évaporés out été étudiés, et on a trouvé que, mis à part les effets thermiques, les imperfections dans le film agissent sur la tension totale.

Letter by R. W. Hoffman R. D. Daniels & E.C.Crittenden,Jr. *Proc. Phys. Soc.* **67B**, June 1954 497-500

The Electron Microscopy of Photographic Grains. Specimen Preparation Techniques and Applications 120/III

See Abstract No. : 111/II

An Improved Replica Technique for Electron Microscopy of Paint Films 121/III

See Abstract No. : 186/I

A Simple Method of Preparing Silica Replicas. Advantages Offered by the Cathodic Etching of Specimens 122/III

See Abstract No. : 113/II

31 — CATHODIC SPUTTERING — 31

Sputtering at Low Ion Velocities 123/III

United States. Arising out of the problem of determining the threshold voltage for sputtering the authors developed a sensitive method for measuring sputtering at ion velocities in the region of 100V. The principle of the method is to measure, during deposition and by means of a probe, the change in collection of electrons furnished by a plasma. The apparatus consists of a bell-jar housing a tungsten helical cathode, a sputtering electrode (negative with respect to the plasma during operation), a cylindrical molybdenum anode, coaxial with the cathode, and a tungsten probe. The latter is movable and can be placed 5–10 mm. away from the sputtering electrode. The difference in the work function of the probe before and after sputtering is a measure of the thickness of the sputtered deposit. The authors discuss several possible sources of error, such as general contamination of the probe from the system resulting in a creep of the value of the work function of the probe. This has to be overcome by a strict and thorough baking schedule. Another source of contamination of the probe is the hot cathode. Although the cathode is made from tungsten this contamination may become appreciable as the temperature of the cathode is 2,400°K and experimental runs take up to 6 hours. Further, care must be taken that the probe does not assume temperatures above a critical level which may induce surface migration of the deposit and in the worst case re-evaporation. The optimum operational pressure for such measurements is governed by a number of factors enumerated in the article, but in experiments sputtering platinum in a xenon atmosphere this pressure was determined practically to be between 20 and 30 micron Hg. This is the region where ion current tends to depart from a constant value. A typical cycle of measurement is: (1) Characteristics of the clean probe, (2) period of sputtering, (3) characteristics of the covered probe and (4) characteristics of the freshly cleaned probe. Sputtering at 200 V for 5 seconds produced a change in the work function of 0.1V. At 150 V the same increase in work function was obtained after 10 seconds, at 100 V after 34 seconds and at 40 V after 5,400 seconds.

Sommaire : Des taux minimes de projection cathodique sont mesurés, au moyen d'une sonde plaçée dans la décharge.

Article by G. Wehner & G. Medicus *J. Appl. Phys.* **25**, June 1954 698-702

III — Vacuum Processing Techniques — III
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Abstract No. and References

The Optical Absorption Constant of Cadmium Oxide

124/III

Germany. Reference is made to work published by Helwig on the electrical and optical properties of cadmium oxide layers prepared by cathodic sputtering. Helwig showed that both these properties depend largely on the methods of preparation and on the thermal case history of the films. Films sputtered in an oxygen-poor, oxygen-nitrogen atmosphere transmit blue-green light, whereas films treated in a pure oxygen atmosphere transmit only yellow and red light. This change of absorption property, *i.e.* the increase of the value of the absorption constant, is attributed to the order of magnitude of self-absorption in the green range of the spectrum. It is the purpose of the investigation reported here to clarify the reasons for the change in the optical properties in that range of the spectrum. The author prepared the films to be examined by the same method as described by him previously (*Z. Phys.*, **134** (1953), 194). As indicated in Helwig's experiments it was found that the actual region where the absorption constant values rise rapidly and lead to self-absorption, *i.e.* the absorption wedge, at room temperature depends very largely on the method of the preparation of the film and its thermal case history. An annealing treatment will shift the absorption wedge and differences as high as 0.5 eV may be found However measurements show that not the whole of the absorption curve is shifted. In fact only the extreme end of the self-absorption is shifted and no changes occur in the values of the absorption constants in the remainder of the self-absorption region. Electrical conductivity and region of the absorption wedge are closely related at room temperature. As the conductivity increases the position of the absorption wedge moves towards the region of shorter wavelengths. The temperature dependence of the value of the absorption wedge is the same as in the case of most other semi-conductors. At high temperatures it moves in the direction of the region of long wavelengths. Two tentative explanations are given for this behaviour.

Sommaire : Les films d'oxyde de cadmium possède une bonne conductivité et une valeur de la constante d'absorption qui n'est pas très differente de celles des semi-conducteurs. Les propriétés optiques semblent dépendre de la méthode de déposition de la couche.

Article by J. Stuke *Z. Phys.* **137**, No. 4 1954 401-415

A Simple Method of Preparing Silica Replicas. Advantages Offered by the Cathodic Etching of Specimens

125/III

See Abstract No. : 113/II

33 — IMPREGNATION — 33

Silicate Impregnation of Porous Castings

126/III

United Kingdom. It has been proposed in the past to employ a sodium silicate solution containing potassium dichromate for the impregnation of aluminium and magnesium base alloys, a process frequently employed to minimise porosity. It has been found that such a solution contains an alkali radical which attacks aluminium. In order to overcome this difficulty it is suggested to add chromic acid to the solution. The preferred solution has the following composition: Chromic acid 7% by weight, potassium dichromate 5% by weight and the balance water, water glass 50 parts by volume. The latter has a sodium silicate content of about 1 part by weight sodium oxide and 2 parts by weight silicon dioxide. Processing details are given as follows: The castings are placed into a vacuum autoclave which is evacuated for a period of 30 minutes at a pressure of no less than 25 inch. Hg. Then the impregnating solution is admitted at a temperature of about 95–100°C and positive pressure of 60–80 p.s.i. is applied for about 30 minutes. After completion of this treatment the castings are removed, washed and heated for about 1 hour at 120°C.

Sommaire : Description d'une méthode d'imprégnation sous vide des moulages d'alliages à base d'aluminium et de magnesium, employant une solution de silicate de sodium contenant du bichromate de potassium et de l'acide chromique pour le traitement.

L. E. Probert, W. A. Sutton F. Cork & Rolls-Royce Ltd. *U.S. Pat.* 2,680,081

36 — DRYING — 36

True Fruit Flavours

127/III

See Abstract No. : 149/III

Stabilised Orange Juice Powder I. Preparation and Packaging

128/III

United States. The preservation of concentrated orange juice in the form of powder obtained by a puff-drying process has been investigated. The feed material most suitable for the process is high density concentrate containing up to 65% soluble solids as can be obtained from plants producing frozen concentrated orange juice. A layer of this concentrate, 1/16th inch deep, was held by trays of a vacuum shelf dryer. The material in the trays was preheated to a temperature not exceeding 38°C during the pumping-down time which took about 4 minutes (10 mm. Hg). When operational pressure was reached the temperature was raised to 200°F but due to the rapid evolution of water vapour the temperature dropped to about 35°F in the first ten minutes before it began to rise again. At this stage an appreciable evolution of gases took place which, because of the

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viscosity of the concentrate, caused the concentrate to swell instead of spatter. During this phase which lasted 20 minutes the temperature did not exceed 100°F but it tended to rise after the puffing phase which would lead to overheating if left uncontrolled. After total drying time of 90 minutes moisture content was about 3%. Final shelf temperature should be 140°F. During the process the feed grows to a thickness of about 2 inch. The trays are silicon-coated to facilitate easy removal of the product. The end product of drying is crushed into a powder of about 20 mesh which has a bulk density of 0.3 g./ml. and can be easily reconstituted. Finer powders do not reconstitute equally well. The pressure during the process should preferably be kept uniform at 5 mm. Hg. The addition of low-conversion spray-dried corn-syrup solids (24 dextrose equivalent) resulting in a feed having 60% orange solids and 40% corn-syrup solids increases the readiness of the material to puff. Flavour was added to the finished powder by admixing sorbitol–oil granules just before packaging. Details of the preparation of the sorbitol–oil mixture are given. To keep the cost of the process low, in–package desiccation is used to remove the residual moisture from the material once it has been packed in hermetically sealed containers. The most suitable desiccant material is calcium oxide or calcined lime which is held in a sift-proof moisture–permeable paper bag and placed in the container prior to sealing. An amount of desiccant equal 10% of the weight of the orange powder in the can will keep the orange powder substantially at zero moisture content. Experiments with a continuous belt-type vacuum drier have indicated that orange powder puff-dried by the method described can be produced with such equipment under commercial conditions.

Article by S. I. Strashun & W. F. Talburt *Food Technol.* January 1954 40-45

Sommaire : Description de nouveaux développements apportés dans la préparation, la stabilisation du parfum et l'emballage de poudre de jus d'orange.

Stabilised Orange Juice Powder. II. Changes During Storage

129/III

United States. Orange juice powder prepared by the puff drying method described in Abstract 128/III has been tested to determine the factors which influence its stability during storage. All material tested was packaged as described before using in-package desiccation with the help of calcined lime as the desiccant. 750 samples were employed in the investigation to study the effects of atmosphere, storage temperature, type of orange oil, method of adding oil, in-package desiccation and addition of sulphur dioxide. The samples were packed in enamelled cans (211 × 414), each can containing 170 g. of powder. The cans were sealed in a semi-automatic double seamer which was fitted with a vacuum chamber which was used for vacuum packaging at a pressure of 29 inch. Hg. After packing, controlled samples were held at –30°F and the others were stored at 70°F for a conditioning period of 75 days. During this period the moisture content of the sample was reduced from its initial 3% to 1.4 and 1.7% for vacuum and air packed samples respectively. After conditioning, groups of samples were stored at temperatures of 70°F, 90°F and 100°F and were examined at regular intervals. The moisture content of the powder was found to be the most important factor governing storage life. Moisture content should preferably be reduced below 1% during the conditioning period before storage at high temperatures. Stability was effected by the method of incorporating orange oil in the powder, the addition of orange oil in sorbitol being more satisfactory than the direct addition of oil. Stability was improved by the addition of small quantities of sulphur dioxide to the powder. Vacuum packing resulted in slightly improved storage stability. A general survey of the results is given in a table partly reproduced below.

Panel Scores of In-Package Desiccated Samples of Orange Powders Stored at 70°F (21°C) and 100°F (38°C)

Type of Sample	*Organoleptic Rating*				
	*Storage at 100°F**			*Storage at 70°F*	
	60 *days*	120 *days*	180 *days*	180 *days*	360 *days*
Cold-pressed oil in sorbitol:					
Sulfite added, vacuum pack	7.7	7.4	6.9	8.4	8.2
No sulfite added, vacuum pack	6.7	6.1	6.5	8.2	8.1
Terpeneless oil added directly to feed:					
Sulfite added, vacuum pack	7.1	6.8	6.8	8.0	7.4
No sulfite added, vacuum pack	6.7	5.5	5.5	6.9	7.0
Sulfite added, air pack	6.2	6.1	6.4	7.5	7.2
No sulfite added, air pack	5.5	5.2	6.0	7.3	6.8

*Samples stored 75 days at 70°F prior to placing in storage at 100°F; rating at 10 is highly palatable; 6 is the limit of palatability; and 1 is extremely unpalatable.

Article by A. M. Mylne & V. S. Seamens *Food Technol.* January 1954 45-50

Sommaire : Rapport sur les résultats d'une étude de stockage de poudre d'orange contenant des sirops de céréales séchés sous vide.

Apparatus for Stirring a Reaction Mixture Through a Vapour-Tight Seal

130/III

See Abstract No. : 125/II

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Abstract No. and References

37 — METALLURGICAL PROCESSES — 37

Principles of Vacuum Distillation of Metal Mixtures 131/III

See Abstract No. : 195/I

Still 132/III

United States. The design of a molecular still is described particularly suitable for producing and separating UCl_4 and UCl_6 from UCl_5. Essentially the still consists of two cylindrical outer casings, each closed at one end and assembled by means of a flange connection in such a manner that they form a fully closed cylinder. The seal between the two casings is made airtight by two ring-shaped gaskets designed in such a manner that leak-tightness can be tested by connecting an annular space between them to a vacuum pump and suitable means of indicating a leak. A hood-shaped heater can be lowered over the assembly to cover the upper casing; the lower casing is surrounded by a cooling coil. The upper casing holds a cylindrical container, closed at the bottom, with various vertical partitions which are open at the top. The wall of that container is recessed on one side so that a passage is formed between the upper and lower casing of the still, facilitating the circulation of gases or vapours between the upper and the lower sections. The lower casing is connected to a vacuum pump and is fitted with baffles in such a manner that no vapours can escape into the vacuum line. The production of UCl_4 and UCl_6 in the still is carried out in the following manner: A charge of UCl_5 is placed in the compartments of the upper container. The still is assembled, hermetically sealed and evacuated to a pressure of 10^{-4} to 10^{-5} mm. Hg. Cooling water is circulated through the cooling coil to give a wall temperature of 40°C in the lower section. The heater is lowered on to the upper casing and the walls of the upper casing are heated to about 200°C. As a result UCl_5 is decomposed to UCl_4 and UCl_6. The former remains in the upper container as residue, while the latter is sublimed because of its relatively high vapour pressure and circulates to the lower section where it is condensed and collected. When the production of UCl_6 vapours ceases, the process is complete. The heater is removed, pumping is stopped and the cooling water is turned off. The still is transferred to a cabinet, containing carbon dioxide or a suitable inert gas, for unloading and separate bottling of UCl_6 and UCl_4. The still is designed to accommodate a charge of 2,200 g. of UCl_5. One run normally takes 14 hours. The yield obtained is about 1,007 g. UCl_4 and 1,193 g. UCl_6. Further applications of the still, in particular in connection with the purification of UCl_4, are given.

R. J. Schmidt S. Rosenfeld & U.S Atomic Energy Comm. *U.S. Pat.* 2,682,451

Sommaire : On décrit l'exécution d'une colonne de distillation du type moléculaire particulièrement utile pour la production et la purification du chlorure d'uranium.

Diffusion of Hydrogen in Mild Steel 133/III

See Abstract No. : 220/I

An Improved Method of, and Apparatus for, Determining Gases Contained in Metals 134/III

Austria. Metals when heated liberate gases. The determination of the quantity of the gases evolved normally requires the use of high vacuum apparatus comprising mechanical and diffusion pumps. A method is described for the determination of the gases liberated by the hot extraction method which does not require high vacuum pumps of the type mentioned. Instead, a simple mercury air pump is used of the type which produces a pumping action by raising and lowering a mercury container. Essentially the method consists of introducing the specimen to be tested into a quartz tube closed at one end and fitted to a suction line at the other. A small electric oven, moving on rails, can be placed over the closed end of the quartz tube. The suction line is connected to the mercury air pump at a point some 80 cm. below the test tube. A further 80 cm. lower, a gas collecting vessel is situated, the connecting pipe leading in to it, being immersed in mercury contained in that vessel. A side tube joins the suction line near the point where the latter joins the quartz test tube. At the bottom of the side tube is a small container holding an absorbent, such as active carbon or dehydrated chabazite. The vessel holding the absorbent can be cooled by moving a Dewar flask into a position surrounding it, the Dewar flask being filled with liquid air. After introducing the specimen into the test tube and connecting the test tube to the suction line, the whole equipment is evacuated by means of the mercury air pump and the oven is placed in position to heat up the specimen. At the same time the Dewar flask is raised to cool the absorbent. As a result of the gases evolved from the specimen being absorbed by the absorbent, the vacuum in the system is maintained without further pumping. When all gas has been collected the oven is removed, the Dewar vessel is lowered, and the gases coming off the absorbent are pumped into the gas collecting vessel. The same pump can be used to transfer the gases from the gas collecting vessel to an analyser after the appropriate connections have been made. The apparatus described is designed for the testing of steel specimens for hydrogen content at temperatures up to 1,200°C. The apparatus can be used for tests at higher temperatures but it requires certain modifications which are described in detail.

Ver. Oester, Eisen und Stahl Werke A.G. *Brit. Pat.* 706,535

Sommaire : Description d'un appareil comparativement simple et bon marché pour analyser des gas tels qui ceux libérés pendant le chauffage d l'acier.

Precision Determination of Low Concentrations of Carbon in Metals 135/III

United States. Many methods for the determination of carbon concentrations in metals lower than 0.01% require expensive high vacuum equipment or elaborate oxygen purification systems. A simplified method is

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described which eliminates the use of high vacuum pumps. The equipment employed consists of two sections, one operating at near-atmospheric pressure and the other operating at reduced pressure, obtained by a mechanical pump. The former section consists of a standard Lindberg high frequency combustion unit, a gasometer (a standard Lindberg gas buret), a levelling bottle and levelling solution which consists of sodium sulphate plus sulphuric acid. A 4-way stopcock connects to the low-pressure system which incorporates a liquid nitrogen trap, a McLeod gauge and a safety valve in the line to the vacuum pump. The 'atmospheric' section is flushed before the first, and between all subsequent runs, with oxygen. The sub-atmospheric section is pumped between the runs at a pressure of 2 micron Hg, flaming the cold trap during the process. A 1-g. sample, placed in a standard Lindberg 'cuplet', is inserted into the combustion unit, the levelling solution is fed into the gasometer and the combustion is started. During this phase the atmospheric section is shut off from the reduced-pressure section. The combustion products collect in the gas buret and the progress of the combustion is adjusted in accordance with the movement of the level of the solution observed in the buret. After completion of the combustion, the pump evacuating the sub-atmospheric section is shut off and the McLeod gauge is isolated. Subsequently the gases are transferred from the buret to the cold trap. After two minutes, allowed for the condensation of the gases in the trap, pumping is re-started to remove the oxygen. While pumping continues, the stopcock controlling the McLeod gauge is opened. After about ten minutes pumping is stopped and the liquid nitrogen in the Dewar vessel, surrounding the trap, is replaced by an alcohol slush, a mixture of methanol containing a few millilitres of water and liquid nitrogen, which has a temperature of –102°C. This causes the carbon dioxide to sublime into the measuring volume where the pressure is determined with the McLeod gauge. The accuracy of the method is indicated in a table partly reproduced below.

Sample Designation	*No. of Determinations*	*NBS Value* %	*KAPL Value* %
NBS 166	5	0.0274	0.0268
NBS 131	10	0.0028	0.0028

NBS = National Bureau of Standards standard sample.

The new method permits operation at temperatures of 1,700°C plus, whereas conventional low-pressure systems cannot be operated above 1,300°C. Further, the excess oxygen, present when the gases are transferred to the trap, causes an appreciable increase of the transfer rate. As a result of the condensation of the excess oxygen in the trap the carbon dioxide is 'carried' to the trap in a manner analogous to the action of a diffusion pump and the transfer rate is no longer dependent on the temperature gradient only. The method has been employed for carbon determinations in iron and steel, manganese, molybdenum, copper, lead, titanium and tin.

Sommaire : Description d'une méthode simple et précise pour la détermination de petites concentrations de carbone dans des métaux.

Article by L. P. Pepkowitz & W. D. Moak *Analyt. Chem.* **26**, June 1954 1022-1025

Vacuum-Fusion Analysis of Molybdenum

136/III

United States. A vacuum-fusion apparatus was built which is capable of giving results reproducible to within ± 1 p.p.m. by weight for both oxygen and nitrogen. The use of mercury cutoffs and all-glass diffusion pumps and furnace envelope reduces the volume of blank gases to 0.006 c.c. per hour at 1600°C. With the sensitivity of analysis provided by this apparatus, the effects of minute quantities of oxygen and nitrogen on the physical behaviour of molybdenum may be determined.

(*Nuclear Science Abstract*)

Sommaire : Un appareil de fusion sous vide, capable de donner des résultats reproductibles à ± 1 p.p.m. par charge pour oxygène et azote.

Article by M. W. Mallet & C. B. Griffith *Trans. Am. Soc. Metals* **46**, 1954 375-388

Furnace Spectrum of Plutonium

137/III

See Abstract No. : 197/I

The System Uranium-Mercury

138/III

See Abstract No. : 196/I

Arc Melting Kroll Zirconium Sponge

139/III

United Kingdom. Zirconium sponge, the end product of the Kroll process for the production of zirconium metal, requires further treatment to remove impurities and to consolidate the metal before it can be fabricated in the form of sheet, bar, etc. The arc melting method is the most suitable process for this treatment as it obviates the use of crucibles. A detailed description is given of an arc melting method recently developed for the purpose by the U.S. Bureau of Mines which covers the production of zirconium and zirconium alloy ingots. Processing in a rare gas atmosphere will not eliminate all impurities. For instance, in order to remove hydrogen, it is necessary to re-melt the ingots in a vacuum arc. Reference is made to practices in titanium production.

Sommaire : Un donne une déscription détaillée d'une méthode de fonte par arc développée par le Bureau Des Mines U.S., pour le traitement de 'sponge' de zirconium.

Article by G. L. Miller *Vacuum* **4**, April 1954 168-175

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Sintered Thermionic Cathodes Formed from Nickel and Alkaline Earth Oxides — 140/III
See Abstract No : 59/IV

Studies on the Creep Recovery and Annealing of Zinc Single Crystals — 141/III
See Abstract No : 177/I

38 — DISTILLATION — 38

Still — 142/III
See Abstract No : 132/III

High-Vacuum Fractional Distillation Column — 143/III

United States. The apparatus is equipped with a vacuum pump producing an absolute pressure of 0.001 to 5 mm. Hg. It is successfully used in the purification of hexadecane with octadecene to obtain a product having 98.8% purity. Crude diallyl phthalate was distilled to an acidity of 0.04 meq. per 100 g. and a colour of 17 (Pt–Co scale).

(*Chemical Abstracts*)

H. Dannenberg & Shell Development Co. *U.S. Pat.* 2,671,053

Sommaire : Description d'une colonne de distillation à basse pression pouvant être employée pour la purification de $C_{16}H_{34}$ par du $C_{18}H_{38}$.

High Vacuum Distillation and Drying System — 144/III

United States. It is proposed to improve the efficiency of short-path vacuum stills, where the evaporating and condensing surfaces are within a few mean-free-path lengths of each other, by several devices. Firstly, a 'carrier' gas, such as carbon dioxide, is introduced into the vacuum system and directed from the evaporator to the condenser. A gas must be chosen for the purpose which will not react with the liquid or the vapour phase of the other materials present in the still nor should it contaminate the distillate. The gas is continuously removed from the system by the action of the vacuum pumps, purified and re-circulated. Although the introduction of a gas into a high vacuum still in this way is contrary to normal practice, it can serve to assist the transfer of the vapours, rising from the surface of the liquid, to the condenser. Jets may be provided above or below the surface of the liquid so that the carrier gas either enters the vapour stream direct or stirs the liquid before it enters the vapour. Stirring will have the effect of reducing surface concentration of non-volatile fractions in the distilland. Secondly, it is proposed to introduce a low-boiling point liquid, such as ether, into the distilland, if the latter is particularly heat-sensitive. As a result the boiling point of the evaporating material is reduced and this facilitates the recovery of heat-sensitive compounds at substantially lower temperatures. The liquid chosen for the purpose must be immiscible with the processed material and should not react or condense in the still. As in the case of the carrier gas, the liquid may be purified after removal from the system by the vacuum pumps and re-circulated. Thirdly, it is suggested to use a conical condenser, apex upward and rotating about its own axis. This arrangement causes the distillate to move across the surface of the condenser under the action of centrifugal force. When the distillate reaches the edges of the condenser, it will be thrown off and collect on the walls of the still. The latter are maintained at a temperature which prevents re-evaporation of the distillate or condensation of its light ends. A still incorporating the devices described has been employed with success for the removal of tocopherols from the by-products of the glyceride oil deodorisation process.

B. H. Thurman & Kraft Foods Co. *U.S. Pat.* 2,682,499

Sommaire : On propose d'augmenter l'efficacité de distillateurs sous vide du type 'short path', en introduisant un gaz porteur pendant l'opération.

39 — MISCELLANEOUS PROCESSES — 39

Air Pump for Food Containers — 145/III
See Abstract No. : 96/II

Flexibles are Re-Vamping Package Pattern — 146/III

United States. Developments in the field of flexible-type food packaging are reviewed. Reference is made to the use of pouches made of Cellophane-Plio film for packing sliced bacon and luncheon meat in vacuum. The method ensures that the meat keeps fresh for long periods and discoloration during display is prevented. Cheese packed in this way keeps moist but mould-free for over a month. Various machines are mentioned vacuumising and heat-sealing pouches semi-automatically. In one case the machine occupies a floor space of only 4 ft.^{2} and

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handles 2.160 packages per hour. Certain soft foods, *e.g.* cheese, cannot be vacuum-packed in slices because the slices tend to 'weld' together. This difficulty may be overcome by inert-gas packaging. Sliced foods are hand-packed into pre-formed pouches which are then automatically vacuumised and sealed. Machines are available to pouch-pack ice cream sundae toppings at a rate of 60 pouches per minute. Saran-type copolymer films are used for making skin-tight bags for products such as poultry, meat and ham. The whole sequence of operations, *i.e.* shrinking, sealing with aluminium clips and vacuumising is carried out on one production line giving a continuous output. Developments in inert-gas packaging are expected to facilitate in the near future the use of pouches which tolerate steam-sterilisation, promising materials being a heavy-gauge vinyl film and a vinyl-foil laminate. Another problem investigated at the present moment is the sterilisation of packaged food by means of ionising irradiation.

Sommaire : Etude des méthodes d'emballage sous vide appliquées à l'emballage des aliments.

Article by J. V. Ziemba *Food Engng.* **26**, March 1954 70-74, 126, 128

Stabilised Orange Juice Powder. I. Preparation and Packaging — 147/III

See Abstract No. : 128/III

Stabilised Orange Juice Powder. II. Changes During Storage — 148/III

See Abstract No. : 129/III

True Fruit Flavours — 149/III

United States. The best flavour quality is obtained from fruit processed immediately it is picked. However, as a rule processors are situated in areas remote from the food-growing districts and rely therefore on supplies in a preserved condition. Such preservation is normally effected by freezing the fruit. The method of extracting juice depends on the nature of the fruit. Apples and grapes may be simply crushed and pressed but citrus juices must be expressed in such a way that the bitter peel is eliminated. Pine-apple juice requires a special method and is deaerated under high vacuum. Methods of preserving the juice vary. Pasteurisation is applied for the preservation of grape, apple and pine-apple juices but freezing is the preferred method in the case of the heat-sensitive citrus fruits. Impregnation with carbon dioxide to about 110 lb. pressure is used in Europe for preserving apple juice. Concentration by freezing yields the best quality product but is expensive and permits only low concentrations. The method is normally applied only to the processing of citrus fruits and strawberries. In the case of other juices a combination of freezing and evaporation may be used or vacuum evaporation only. The latter is carried out at a vacuum of at least 28 inch. which reduces the boiling point to a value, where caramelisation of fruit sugar is prevented. In this method, however, some of the volatiles which contribute to flavour and aroma are lost. Various processes are described designed to prevent the loss of these volatiles. According to one method, the Serailian process proposed in 1917, the juice is concentrated in a vacuum pan and the vapours formed in the process are fractionated. The fraction containing the highest concentration of volatile aroma is returned to the concentrate. Another method, developed by Pfaudler 10 years later, belongs to the same class of processes. Details are given. A different type of process has been developed by the Eastern Regional Research Laboratories of USDA in 1944. Here the respective volatiles are stripped from the juice at an early stage of the concentration process and are returned in the form of 'essence' to the final concentrate, obtained from the remainder of the juice. In conclusion the author discusses the merits of concentration *versus* single-strength juice. Concentrates are preferable because they facilitate standardisation of the quality of the product in terms of flavour strength, colour and acidity, storage is more economical and true fruit flavouring may prove cheaper than single-strength juices. However, imitation flavours may be applied with advantage, where the product is subjected to intense heat involving a high loss of flavour. A table is shown giving the types of flavours which produce best results in various foods and beverages.

Sommaire : Discussion sur les méthodes de préservation et de recupération du véritable parfum dans le traitement des jus de fruit.

Article by P. Adams *Food Engng.* **26**, June 1954 93-94, 97, 129-130

Vegetable Cooling by the Vacuum Method Process — 150/III

United States. Vacuum cooling of leafy vegetables, in particular lettuce, was first carried out on a commercial scale in 1946 but it became generally acceptable only in 1950. In that year the company pioneering the vacuum cooling process introduced a new method of packing lettuce in partially ventilated cartons which greatly helped acceptance of the process by the trade. Today there are several plants in the U.S.A., one of them capable of handling 95 car loads of lettuce in 22 hours. Transportation on the plant's premises is assisted by the use of large-fork lift trucks lifting whole van loads of lettuce, conveyor belts and two-way radio communication between the crews. One vacuum chamber can hold 14,400 lb. of lettuce having a dimension of 7½ ft. width and 51 ft. length. Actual cooling takes 20 minutes per charge. Preliminary evacuation is carried out by a two-stage steam ejector, while a three-stage steam ejector takes over during the cooling period handling up to 860,000 cu. ft. of vapour during that period.

Sommaire : Un article détaillé sur les progrès réalisés dans l'application de la congèlation sous vide des legumes à grandes feuilles.

Article by W. A. Webb *Industr. Refrig.* **126**, May 1954 15-18

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Curbing Jack Frost

151/III

United States. Trouble is frequently experienced when a sudden drop of pressure occurs in a buried gas pipe producing a refrigeration effect, which results in frost forming in the adjacent ground and causing it to heave. In order to protect the ground the gas pipe was enclosed with a larger pipe (16 inch. diameter in the case of a 6–inch. gas pipe) for a distance of 30 ft. downstream from the point of the reducing valve. The larger pipe was sealed at each end and evacuated. This produced satisfactory thermal insulation.

Note by Anon. *Compr. Air Mag.* April 1954

Sommaire : Le vide est employée comme isolément thermique là ou les effects de refroidissement, causés par une soudaine chute de pression dans les conduites de gaz enterrées, causent un soulèvement du sol.

Light-Transmissive Electrically Conducting Optical Article

152/III

See Abstract No. : 117/III

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40 — GASES AND VAPOURS — 40

A Graphical Approach to Vacuum Engineering 38/IV

See Abstract No. : 137/I

Specific Gravity of Gases 39/IV

United States. Six methods of measuring the specific gravity of a gas are described. The first two give direct readings, are basic methods and require the use of vacuum equipment. The remaining four need calibration. (1) *The Direct-Weighing method* is a laboratory method. The weight of a globe filled with air is compared with the weight of the same globe filled with the gas under test at the same pressure and temperature. The apparatus consists of two globes of equal size, one of which is permanently evacuated and acts as the counter balance to the other, the weighing, globe. First the weighing globe is evacuated, its own weight is determined and the balance between the two globes is adjusted accordingly. Then, the weighing globe is filled with the gas to be tested at a pressure as near as possible to atmospheric pressure. When temperature equilibrium has been reached, the globe is weighed and the total weight of the sample and the globe is recorded. This is followed by weighing the globe after filling it with air by the same procedure. A sketch is shown of the filling arrangement. The specific gravity can then be calculated from:

$$(W_{gas} \times P_{air} \times T_{gas})/(W_{air} \times P_{gas} \times T_{air})$$

(2) *The Gas Balance method* is based on Archimedes' principle, *i.e.* the weight of a given volume of a substance immersed in a liquid or gas is equal to the weight of the liquid or gas displaced. Specific gravity in this case is indicated by recording buoyancy. Three varieties of the method are discussed: (*a*) The Edwards Balance, (*b*) the Ac–Me Suspension–Type Balance and (*c*) the Anubis Direct–Reading Balance. In the case of the first a float is attached to a balance beam enclosed in an air-tight chamber. The chamber is evacuated, pure air is admitted at a pressure sufficient to move the float indicator to the null-balancing position. Then, the chamber is evacuated again and the gas to be tested is admitted up to a pressure to move the indicator to the marked balancing position. The respective pressure recordings, and manometer and barometer readings facilitate the calculation of the specific gravity of the gas. The Ac–Me balance has a different suspension system and requires larger samples for measurement. The Anubis instrument facilitates the reading of the specific gravity directly from the scale. (3) *The Effusion method* is based on the phenomenon that the rate of flow of a gas through an orifice of given dimensions depends on its specific gravity. A typical apparatus operating according to this method is the Schilling's Bottle. Details of its operation are given. (4) *The Viscous-Drag method* is represented by a description of the Ranarex Gravitometer. The instrument has two chambers, a gas chamber and an air chamber. Each chamber is fitted with a motor-driven fan, moving at constant speed, and a freely rotating impulse wheel. The air and gas admitted to the respective chambers receive a rotating motion by the fan and exert a torque on the impulse wheel in each chamber. As the fans rotate in opposite directions the torque exerted on the impulse wheels oppose each other. The shafts of the impulse wheels are linked mechanically and the differential of the two torques is indicated by a pointer. The instrument is suitable for continuous recording and requires 6 ft.^{3} p.h. of approximately atmospheric pressure gas for its operation. (5) *The Kinetic-Pressure-Increase method* is represented by a description of the Metric Gravitometer and (6) *the Air-Column-Weighing method* by a description of the Anubis Direct-Weight Type of Gravitometer.

Sommaire : Six méthodes de mesure de la gravité spécifique des gaz sont décrites.

Article by B. J. Willard *Instruments* **27,** June 1954 945-949

Ionisation and Excitation of Mercury Vapour by Positive Ion Impact 40/IV

See Abstract No. : 156/I

Composition, Decontamination of Radioactive Gas Mixtures 41/IV

United States. In the operation of a 'Water-Boiler' reactor the uranyl–nitrate solution in natural water is radiolytically decomposed resulting in the liberation of hydrogen, oxygen, nitrogen and fission products which mix with the air flushed through the system to keep the radioactive gas away from the pressure gauge lines. In a recombination system hydrogen and oxygen is converted into water which is returned to the solution in the reactor. A description is given of a micro-volumetric system facilitating the analysis of the 'off-gas' mixture. This gas mixture is highly radioactive because of the presence of products of the iodine-bromine decay chain such as xenon and krypton. Iodine, for instance, constitutes a serious health hazard and therefore the limit of 8–day I^{131} permissible in breathable air has been set at 3×10^{-9} mc /l. Thus only very small samples can be used. The all–glass apparatus used for the analysis is shown in an illustration. Mercury cut-offs are employed throughout, instead of mechanical valves. A mercury diffusion pump is used for evacuation. The gas sample to be analysed is supplied in closed tubes which are sealed to the storage bulb of the system. A 200–ml. antomatically controlled Toepler pump is employed for direct transmission of the sample to the 50–ml. measuring bulb, or, alternatively, for circulation of the sample over the reagents used for the removal of its individual components. The determination proceeds in the following order: Carbon dioxide plus moisture by freezing with liquid nitrogen, moisture after separation from carbon dioxide by fractional sublimation, hydrogen by combustion with oxygen, oxygen by oxidation of hot copper, nitrogen by reaction with hot uranium turnings and inert gases by difference. Details of the procedure are given. It follows a description of the method employed

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for the collection of the gas samples to be analysed. An indication of the radioactivity of the sample when collected is given in the table reproduced below.

Radioactive Gases from Fission with Half-Lives Greater than One Minute

Br^{82}	35 h	Kr^{88}	2.8 h	I^{136}	1.4 m
Br^{83}	2.4 h	Kr^{89}	2.6 m	Xe^{131}	12 d
Br^{84}	33 m	I^{129}	10^8 y	Xe^{133}	5.27 d
Br^{85}	3 m	I^{131}	2.0 d	Xe^{135}	13 m
Kr^{83}	1.9 h	I^{132}	2.4 h	Xe^{135}	9.2 h
Kr^{85}	4.4 h	I^{133}	22 h	Xe^{137}	3.9 m
Kr^{85}	9.4 y	I^{134}	54 m	Xe^{138}	17 m
Kr^{87}	78 m	I^{135}	6.7 h		

O The total intensity at the time of collection exceeded 1 c./ml. N.T.P. The collection system contained a 1-litre receptacle where the gas was left to decay for 2 days before it was transferred to the analytical system. Its activity was then reduced to 20 mr./ml. All valves in the collection system were remote-controlled and pressure measurements were taken from a distance of 2 metres. The whole of the collection system was shielded with 8-inch lead. The results of a typical analysis are given in tabular form. Finally three methods for the further decontamination of the gas (after use in the analysis) are described. The first provides for a slight delay and a large dilution by air in a stack. The second relies on the use of delay tanks where the gas mixture is left to decay for a considerable time. Numerical values are shown in graphs. The third method consists of passing the radioactive gas stream over activated charcoal which absorbs the radioactive components. 100 kg. of charcoal per month would be adequate to effect decontamination of the gas stream evolved from a water boiler operated at a maximum power of 30 kW for 6 hours every day.

Sommaire : On discute les méthodes d'analyse des gaz radioactifs liberés par la decomposition de nitrate d'uranyl qui se fait dans une chaudière de réacteur.

Article by H. M. Busey *Nucleonics* 12, May 1954 9-13

Determination of Oxygen in Certain Gases

42/IV

United States. Present-day studies of corrosion of metals by liquids necessitate the preparation and analysis of inert gas blankets free, or nearly free, of oxygen. Micro-amounts of oxygen may be determined in the inert gases, hydrogen, nitrogen, and carbon dioxide by a modification of the Winkler method, in which the oxygen is absorbed in manganous hydroxide; an equivalent amount of iodine is liberated, then extracted into *o*-xylene and determined colorimetrically. The analysis takes place at low pressures, from 350 to 760 mm. Hg, whereby a small volume of gas (250 to 500 millilitres) can be analysed. A reproducibility of 0.7 parts per million or better was obtained over a range of 0 to 25 parts per million of oxygen (microlitres per litre). The method can be extended to 150 parts per million of oxygen by extracting into larger quantities of *o*-xylene. Improved methods of removing dissolved oxygen from analytical reagents result in a constant, but low, blank which permits the degree of precision obtained.

(*Nuclear Science Abstracts*)

Report by L. Silverman and W. Bradshaw *AEC. Rep. No. NAA-SR-892* April 1954

O *Sommaire* : On donne des détails sur la détermination de micro-quantités d'oxygène dans des gaz inertes, hydrogène, azote et oxyde de carbone, par la méthode modifiée de Winkler.

Determination of Low Concentrations of Oxygen in Hydrogen

43/IV

Canada. In order to facilitate the study of poisoning of an iron–synthetic ammonia catalyst by oxygen an analytical method was required for the determination of oxygen content ranging from 1 to 100 p.p.m. The method developed for the purpose is based on that by Bamford & Baldwin which facilitates determinations down to 0.01% oxygen. The new method has a lower limit of 0.00005% equal 0.5 p.p.m. ± 10% in the case of a 1.3 litre sample of hydrogen. Essentially the experimental equipment consists of a mercury piston pump which circulates the gas over a platinum catalyst; the water vapour formed in the process is passed into a liquid-nitrogen trap for condensation. The volume of the condensate is measured at 10 mm. Hg pressure in a McLeod-type gauge. All calibration data and results are given in litre-micron units. 1.3 litres of hydrogen at atmospheric pressure is 1,000,000 litre–micron and an oxygen content of 1 p.p.m. equals 2 litre-micron of water vapour. Measured at 10 mm. Hg this value is represented by 2 ml. of condensate. Two different water vapour gauges were available, one of 5,500 litre–micron capacity and the other of 150 litre–micron capacity. Details of the plant are given in illustrations. The most critical feature of the method is the amount of water vapour recovered from the gas for measurement. It varies by the amount absorbed by, or desorbed from, the walls (glass) of the equipment. Thus, the temperature of the equipment must be kept constant during the experiment within 1 or 2 degrees. This necessitates the provision of a cooling jacket for the catalyst. In addition, all operational times such as circulation, évacuation and distillation of water vapour must be standardised. A full description is given of the precautions taken to ensure accurate results. Calibration was carried out by adding known amounts of oxygen to dry, oxygen–free hydrogen. The loss due to adsorption of water vapour during measurement depends on the degree of pre–adsorption of water vapour in the gauge. The amount of pre–adsorbed water

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vapour can be determined by evacuating the gauge between two runs. If pre-adsorption is reduced, loss due to adsorption is increased. These conditions are demonstrated in a table, reproduced below.

Recovery vs. Evacuation Time, 5500-Litre-Micron Gauge
Litre-Microns of H_2O

Added, as Oxygen	*Measured, after Evacuating for*	
	5 minutes	*10 minutes*
99.7	66.1	
99.8	67.8	
99.9	68.8	
99.7	67.8	
99.8		61.7
99.8		64.3
99.7		66.3
99.9		65.6
99.8		62.2
99.8	67.0	
Average 99.8	67.5 ± 0.8	64.0 ± 1.7

It is important therefore to keep the pre-adsorption at a high level and in the case of the smaller gauge this was achieved by filling it with 75 litre-micron water vapour prior to an experimental run and evacuating it for 5 minutes. Measurement of the volume of water vapour at 10 mm. Hg is convenient because the pressure is high enough to be read by the unaided eye and yet not too near to the saturation pressure of approximately 25 mm. Hg. Of impurities which would affect the operation of the catalyst only nitric oxide, carbon monoxide and nitrogen may be present. It is assumed that the effects of nitrogen and nitric oxide can be ignored. The presence of carbon monoxide has not been found to be harmful except possibly at very low oxygen concentrations.

Sommaire : Une méthode, basée sur celle developpée par Bamford et Baldwin pour la détermination de concentrations d'oxygène d'environ 0.01 pour cent, permits de déterminer de petites concentrations d'oxygène dans l'hydrogène, de l'ordre de 0.5 p.p.m.

Article by M. M. Wright *Analyt. Chem.* **26**, June 1954 1001-1008

Simple Linde Hydrogen and Helium Liquefiers 44/IV

See Abstract No. : 131/II

41 — FREEZE DRYING — 41

A Freeze Drying Apparatus for Neurohistochemical Work 45/IV

See Abstract No. : 190/I

Apparatuses and Technique for Freeze Drying of Tissues 46/IV

Sweden. Two freeze drying apparatus are described which are essentially of the same design but of different capacity. Both consist of a double-walled cylinder, the outer cylinder being closed at both ends and connected to the vacuum pump, the inner being open at the top to admit the refrigerant and to act as a condenser. The tissue to be dried is inserted into the evacuated cylinder at a point 20 cm. below the bottom end of the condenser in the case of the large apparatus, and 2 cm. away from the bottom end of the condenser in the case of the small apparatus. In the large apparatus the extension of the drying chamber containing the tissue is placed into a Dewar flask and continuously cooled at about –40°C. during the drying process. A mixture of carbon dioxide and alcohol or alternatively liquid nitrogen are used as refrigerants. Evacuation is carried out by a two-stage mechanical pump. Drying chamber and condenser are surrounded by an aluminium sheet and cork packing for the purposes of thermal insulation. The condenser takes about 1 kg. of dry ice. This should be consumed under normal operational conditions at a rate of 0.5 kg. per 12 hours. In the particular design, any appreciably higher consumption of carbon dioxide indicated unsatisfactory vacuum conditions. Best results were obtained when the temperature of the tissue was permitted to rise to about 0°C. towards the end of the drying cycle. The general drying characteristics indicated removal of free water during the first 4 to 6 hours and the removal of bound water during the final period of drying. Liver cylinders (5 mm. × 5 mm.) freeze-dried for 9 hours in the large apparatus using dry ice as the refrigerant, showed a residual moisture content varying from 0.5 to 2%. Fifty specimens of a similar size could be freeze-dried in the large apparatus in 24 hours. Larger pieces were dried for up to 48 hours. All specimens to be dried were pre-frozen either by immersion in propane which remains liquid at –195°C. or by immersion in isopentane at a temperature of –140°C. Both

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apparatus were designed to facilitate the embedding of the dried tissue specimen in paraffin before breaking the vacuum. Results obtained when drying renal tissues are shown in illustrations.

Sommaire : On décrit la construction de deux appareils à lyophiliser des tissus, l'un de grande capacité l'autre de petite capacité, pour travail de routine et pour étudier le temps nécessaire à la lyophilisation des spécimens de tissus.

Paper by B. E. Gustafsson *K. Fysiograf. Sällskapets Handlingar* **64**, No. 15, 1953 1-9

The Application of Freeze Dry Methods to Plant Material

47/IV

United States. Freeze drying methods are widely used in the preparation of animal tissue but with the exception of the work by Goodspeed and Uber little is known about their applications in botanical histochemistry. In order to facilitate proper study the tissue sections prepared for the purpose should retain soluble substances, enzyme activity, undernatured protein and good morphological detail. In the experience of the author many of these requirements are fulfilled by the freeze drying method. The design and performance of freeze drying apparatus for the preparation of plant tissue sections which represents a development of the apparatus employed by Goodspeed and Uber is discussed. 1–4 mm. thick tissue pieces are pre–frozen by immersion in isopentane kept at a temperature just above –170°C. Rapid action is required to prevent the formation of unduly large ice crystals. The segments are then placed into a copper-wire basket and transferred to the dehydration chamber which is connected to a condenser, cooled by liquid nitrogen. The chamber is externally cooled by a dry ice-methyl cellosolve (ethylene glycol monomethyl ether) bath at a temperature ranging from –30 to –60°C. At the bottom of the drying chamber is a layer of paraffin which is melted after completion of the dehydration, permitting the tissue to sink into it and become saturated. The apparatus is evacuated by an oil diffusion pump as a mercury diffusion pump requires the insertion of a special cold trap to prevent back-diffusion of mercury vapours. The provision of a mechanical pump only is sufficient in many cases but invariably extends the period required for drying. Alternative designs of tissue holders are mentioned. The results of exploratory experiments employing the most commonly used species and organs used in morphological and physiological experimental work are given in a table partly reproduced below.

The Segment Size and Dehydration Time Advisable for Various Plant Tissues Based on Results with Common Experimental Species

i.e. Vicia faba, Zea mays, Allium cepa, Lilium longiflorium, Phaseolus vulgaris, Pisum sativum, etc.

Type of Tissue	*Segment size in mm.*	*Dehydration Time in days**	*Remarks*
Leaf	—	1–2	Size of pieces not important except in thick, fleshy leaves (*Sedum, Bryophyllum*).
Stem	1–2–(4)†	2–4	Fairly easy to handle although larger segments have a tendency to tear during freezing.
Stem tip	1–(2)	4–6	Most leaves surrounding tip should be removed before freezing.
Root:			
Mature	1–2–(4)	2–4	Very similar to stem.
With developing secondaries	1–2	4	
Tips	1–(2)	6 plus	Difficult to handle so that there is a minimum of ice crystal formation and tissue separation. Dehydration time can be greatly reduced by using pieces of tissue below 1 mm. in thickness.

* At –35 to –40°C. at a pressure of approx. 10^{-3} to 10^{-4} mm. Hg.

† Parenthesis indicates that satisfactory results were occasionally obtained at this thickness.

The time required for the staining of freeze–dried material is generally appreciably less than that required for material prepared by another method. The quality of morphological detail obtained by this method is shown in illustrations.

Sommaire : On décrit une méthode et un appareil à lyophiliser des tissus de plantes, basés, sur ceux proposés par Goodpseed et Uber, mais plus rapides en ce qui concerne la déshydratation et l'infiltration.

Article by W. A. Jensen *Stain Technol.* **29**, May 1954 143-150

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Preservation of Arterial Grafts by Freeze Drying

48/IV

United Kingdom. A simple method of freeze drying arterial grafts is reported. The grafts are placed into Pyrex test tubes and deep frozen at –79°C. Then the test tubes, closed with sterile gauze, are placed into a glass container connected to a standard penicillin freeze drying condenser by means of a standard glass joint. The unit is evacuated by a single-stage rotary pump *via* a phosphorus pentoxide moisture trap to a pressure of approximately 0.05 mm. Hg. The condenser is filled with a mixture of alcohol and solid carbon dioxide giving a temperature of –79°C. The primary drying cycle in this equipment is short enough to allow for about 6 charges to be dried within a working day. Secondary drying is carried out in a vacuum desiccator which is pumped down initially by a rotary pump for 20 minutes and then kept evacuated for 3 days. The residual moisture content of the artery segments treated in this way was about 2%. After completion of the drying the gauze filters on the test tubes were replaced by rubber caps and the air within the tube was removed by means of a hypodermic needle, attached to a vacuum line, inserted through the cap. On completion of the final evacuation of the tube the hypodermic needle was withdrawn and the test tube submerged and sealed in melted Picein vacuum wax. For the purpose of reconstitution sterile isotonic saline solution was injected through the rubber cap into the tube under exclusion of air. Air was admitted only after the artery had regained normal appearance, *i.e.* after about 30 minutes. Reconstituted arteries had the normal appearance of fresh arteries. The equipment described facilitates the processing of 67 g. of arteries in one batch. Three equal lengths of external iliac artery pre-frozen at –79°C. were subjected to the following treatment: (*a*) Drying without external cooling for 47 hours, (*b*) drying at –30°C. for six days and (*c*) drying at –50°C. for fourteen days. The determination of the residual moisture content in the three specimens indicated that the drying rate was highest in the case of (*a*). Pre-freezing was found to make external cooling during the drying process unnecessary. The effect of freeze drying on the degree of bacterial contamination of arterial grafts has been investigated and the results are shown in the table reproduced below

Quantitative Effect of Freezing and Freeze Drying on Bacterial Contamination of Arterial Grafts

Control culture	*Added bacteria*	*Method of processing graft*	*Result*
10 segments taken aseptically; 7 lightly contaminated with *Staph. albus* (donor with type-II nephritis)	11.8×10^6 viable coliforms between 10 segments	Freeze-dried	Average per segment 340 organisms
10 segments; 7 contaminated with spore bearers (donor with pelvic peritonitis)	8.5×10^6 viable coliforms between 19 segment.	10 Frozen and thawed	3×10^6 organisms
		9 Freeze-dried	7/9 sterile 1 segment 100 organisms 1 segment 1200 organisms
10 segments taken aseptically; 2 contaminated with *Esch. coli* (donor with carcinoma of colon)	75×10^6 *Staph. aureus* between 20 segments	10 Frozen and thawed 10 Freeze-dried	20.3×10^6 organisms 2.1×10^6 organisms

Although the reduction of contamination is satisfactory there is still the need for an aseptic graft-taking technique and careful selection of donors. See also Vol. III Abstract No. 243/I.

Article by H. H. G. Eastcott, L. B. Holt, J. H. Peacock & C. G. Rob
Lancet
26, June 1954
1311-1314

Sommaire : Description d'une méthode simple pour préserver des artères par lyophilisation.

Virus Vaccines Against Rinderpest: The Rabbit Rinderpest Virus. Part II

49/IV

French West Africa. Previous workers have established that the rinderpest virus is particularly sensitive to ambient temperature and must be stored at low temperatures if its virulence is to be preserved. Storing the material at +4°C. its virulence will be maintained for 48 hours. If frozen and vacuum dried, and subsequently stored in a cool but moist atmosphere, its virulence will be preserved for about 1 month. If properly freeze-dried at –20°C. its virulence will be maintained for about 6 months. The results obtained by previous workers are somewhat contradictory but the authors have concluded that freeze drying is the most suitable method for storing rinderpest virus, particularly in areas such as North Africa. A detailed report is given on the use, for the purpose, of the centrifugal freeze drying method as proposed by Greaves, but suitably modified to meet the particular local conditions (Dakar). To prepare the material, virus was injected into rabbits and the animal killed about 60 hours later. Certain organs such as the spleen were removed and pulverised. 4 g. of the pulverised material were mixed with about 20 g. of blood in a Waring Blendor which was pre-cooled. Subsequently 2 c.c. of the suspension were filtered through gauze into each ampoule. The use of physiological serum originally employed as a diluting means was discontinued later as it was found to cause loss of viability in the finished product. The ampoules were placed into the drying chamber and centrifuged. When the cold trap fitted to the system had reached –20°C. the vacuum pump was started. After about 5 minutes a pressure of about 0.1 mm. Hg. was obtained and centrifugation was stopped. As drying proceeded the temperature of the trap reached –50°C. after 2 hours and the pressure reached 35–40 micron Hg. after about 15 hours. The initial cooling-down of the material is due to a self-freezing effect, *i.e.* it results from the evaporation of its moisture content. The temperature characteristics of the processed material covering a full drying cycle are given in a table reproduced on the next page.

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Self Freezing Time	*Ambient Temperature*	*Product Temperature*
0	+ 25°C	+ 26°C
+ 10 min.	+ 25°C	— 20°C
+ 3 h. 30 min.	+ 23°C	+ 8°C
+ 5 h.	+ 22°C	+ 16°C
+ 8 h.	+ 22°C	+ 20°C
+ 16 h.	+ 21°C	+ 21°C

In order to avoid any adverse effects from the high ambient temperature prevailing in that locality primary drying was fixed at 22 hours. In later experiments the additional precaution was taken of cooling the drying chamber for the whole period of the process to a temperature of 2–4°C. Prior to this alteration the residual moisture content of the product after completion of primary drying was found to be 1.3%. Secondary drying is normally carried out at ambient temperatures, but, as in the case of primary drying, provisions were made for the secondary drying to be carried out in a chilled receptacle. Details are given of the method employed to determine the residual moisture content of the finished product.

Article by P. Mornet J. Orue C. Labouche P. Mainguy & R. Mahou *Rev. Prat. Froid* **31**, June 1954 617-622

Sommaire : Une méthode de production de vaccins du virus bovipestique lapiné est décrite, et on discute les problèmes de conservation de ce produit pendant une longue période avant son emploi.

Lyophilisation of Poliomyelitis Virus. Heat Inactivation of Dry MEF1 Virus

50/IV

United States. Few attempts have been made so far to dry poliomyelitis virus. Information available on the behaviour of this virus during and after drying is incomplete. However, in order to measure certain physical properties of the virus it is indispensable to dry the virus first. The authors described a successful method of freeze drying the MEF1 (Type 2) and Leon (Type 3) poliomyelitis virus and record certain experimental observations on their behaviour. The MEF1 baby mouse adapted strain of Casals *et al.* in its 138th passage was used in the experiment. Spinal cords from adult mice infected with the mouse adapted Leon virus in its 90th passage were also used. The virus titrations were done in adult mice by the intraspinal technique of Habel. Details of the preparation of the suspension are given. The material to be processed was deep frozen before use. For freeze drying purposes the material was placed into Pyrex ampoules each holding 0.2 ml. of the suspension which was shell-frozen. The freeze drying apparatus was evacuated by a pump and fitted with a condenser cooled by a mixture of methyl cellusolve and dry ice. Total drying time was 6–8 hours. Originally experiments were carried out at room temperature but at a later stage provisions were made to keep the material cold. Subsequent sealing of the ampoules was carried out in dry air at atmospheric pressure. Various bacteriological media were investigated with regard to their protective effect on dry poliomyelitis virus. It was found that the best survival rate was obtained while drying in the presence of peptone of Na thioglycollate, when the material was reconstituted in water. When the material was dried in a water suspension best results were obtained by reconstitution in 30% peptone. The results of the experiments are shown in a table partly reproduced below.

Results of Drying MEF1 Poliomyelitis Virus in Various Media

% baby mouse brain	*Drying medium*	*Dried in cold*	*Reconstituting medium*	*Virus titer*		*% virus survival*
				Before drying	*After drying*	
20	30% peptone	Yes	water	6.0	5.5	32
10	5% Na thioglycollate	Yes	water	5.0	4.5	32
10	Peptone water	No	water	6.0	4.3	2
20	Water	Yes	30% peptone	6.0	5.0	10

Additional experiments are reported on the heating of dry MEF1 virus. The material sealed in the ampoules after completion of drying was stored at –70°C. until the heating experiments started. In one series the tubes were heated at 10° increments through the range of 0° to 80°C. for a fixed period of 20 minutes. In another series, the material was heated at fixed temperatures *i.e.*, 40, 50 or 60°C. for periods of various lengths. The results of these experiments are shown in graphs. In the first series of experiments no deterioration of the material occurred until the temperature exceeded 20°C. The results of the second series of experiments proved that a heat-resistant variety of the virus was present in the experiments. However at 60°C. the decay became general. The existence of a heat-resistant variety of the virus, indicated in the experiments reported, permits the conclusion that, under favourable conditions in nature, the dry virus may play some part in the dissemination of the disease.

Article by L. M. Kraft & E. C. Pollard *Proc. Soc. Exp. Biol. Med.* **86**, No. 2, 1954 306-309

Sommaire : Il est démontré qui le MEF1 (type 2) tiré d'une petite souris infectée avec du virus poliomyelitique peut être lyophilisé sans perte de sa virulence.

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45 — ELECTRONICS — 45

Design of a Large Vacuum Vessel 51/IV

See Abstract No. : 162/I

Ion Source 52/IV

United States. Normal ion sources, consisting of cold-cathode discharges in a perpendicular magnetic field, are not suited to the requirements of a high current accelerator because they need to be water-cooled and this leads to structural and maintenance difficulties. Also, they are high-voltage, low-current devices. The ion source described here is characterised by its large and relatively intense ion output, small cross-section, high temperature of operation and high overall efficiency. It is designed especially for the production of protons but can also be used as a source of alpha-particles, deuterons and other ions required in particle accelerators. A rectangular carbon block approximately 4 inch. long by $\frac{1}{2}$ inch by $\frac{5}{8}$ inch contains a cylindrical chamber 11/32 inch in diameter, with a slot, $2\frac{1}{4}$ inch. long and 3/32 inch wide, cut into it on the side communicating with the accelerator. A pair of cathodes of tantalum or tungsten 0.050 inch thick and 0.200 inch diameter are placed equidistant from each end of the slot and near to the edge of the slot. Details of the electrical connections are given, the cathode support being joined to a tube which also serves as the gas inlet for the ion chamber. A magnetic field of about 7,000 oersted is situated along the slot parallel to a line connecting the cathodes. In operation, a potential of 1,000 V d.c. is applied with the positive side of the supply earthed, and a glow discharge is initiated between the cathodes. Hydrogen is admitted. Ions formed by collision of electrons with the hydrogen molecules are accelerated along the lines of the magnetic field towards the cathodes. This increases the emission of the cathodes, but as they are made of tungsten or tantalum, they do not overheat, while adding thermally emitted electrons to the general supply. This reduces the potential required to maintain the discharge from 1,000 V to 200 V. The power supply is connected in series with a dropping resistor so that as the arc current increases the potential applied to the source decreases. In spite of reaching high temperatures the source will continue to operate without water-cooling, resulting in a greater electron density than in previous ion sources. Further, the discharge is self-regulating in that any loss of ions to the electrodes causes a drop in arc potential which, in turn, causes the gradient governing the acceleration of secondary electrons to decrease until equilibrium is re-established. The new source produced currents up to 330 mA, 82% of which were protons. By restricting the supply of hydrogen the efficiency of the source can be raised, *e.g.*, an efficiency of 40% could be obtained at an output of only 137 mA, 84% of which were protons. A graph is shown indicating that proton output increases and molecular ion output decreases with rising cathode current.

C. F. Barnett
C. B. Mills &
U.S. Atomic Energy Commission
U.S. Pat. 2,668,260

Sommaire : On donne des détails sur une source d'ions, ayant un débit exceptionnellement large et rélativement intense, et qui semble appropriée pour être employée comme source dans des accélérateurs à particules tel qu'un cyclotron.

Ionisation and Excitation of Mercury Vapour by Positive Ion Impact 53/IV

See Abstract No. : 156/I

Particle Detectors of the Geiger-Muller Counter Type 54/IV

France. The working principle, design and performance of Geiger-Muller counters is discussed. Special reference is made to the types developed at the Commissariat d'Energy Atomique. Basically, a counter consists of a cylindrical cathode of a diameter of the order of one centimetre and a filamentary anode positioned in the axis of the cylinder, the assembly being placed in a receptacle filled with a gas, or gas and vapour mixture, at a pressure of about 1/10 of an atmosphere. The anode is connected across a MΩ resistance to a supply ranging from a few volt to 2 kV. Assuming that two particles pass through the counter one of them liberating 10 ions and electrons in the residual gas, and the other 100 pairs of ions and electrons, subsequent developments will depend on the anode voltage. If the latter is only a few volt, not all the primary electrons will be collected by the anode, due to recombination effects. If the anode voltage ranges between 10 and 200, all charges will be collected, *i.e.* the counter acts as a ionisation chamber. At an anode voltage of 800 the primary electrons will have sufficient energy to cause additional ionisation resulting in an amplification of the original ionisation effect, proportional to the number of primary electrons. This type of counter is called a proportional counter. Raising the anode voltage beyond 1,000, the number of charges collected by the anode is independent of the degree of primary ionisation. This range is called the Geiger zone. The Geiger counter has three significant features: The magnitude of the electrical impulse produced by the primary ionisation is constant and independent of the degree of primary ionisation. Its amplitude being constant, amplification for recording purposes is simple. The liberation of a single electron suffices to produce an electrical impulse. There are two types of counters: (*a*) Requiring external electronic devices to stop the discharge and (*b*) the self-quenching type. The latter is filled with a mixture of a rare gas, such as argon, and an organic vapour such as alcohol. The primary electron created by a nuclear particle traversing the counter will move under the influence of the electric field towards the filamentary anode. Arriving in the vicinity of the latter it will have acquired sufficient energy to cause ionisation by collisions with other gas molecules. As a result an avalanche of electrons is initiated. In the course of this process some of the molecules reach a meta-stable state and will issue quanta of light before they return to their former basic state. The latter are absorbed by the organic vapour present but create photo-electrons during the process which initiate fresh avalanches of electrons. Eventually the whole length of the

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anode is surrounded by an inner sheath of electrons and an outer sheath of positive ions. The electrons will be rapidly collected by the anode and the ions will move in the direction of the cathode. This phase is termed the 'dead' time of the counter and lasts about 0.5×10^{-4} sec. During this period the counter will not register any fresh primary ionisation event. The sheath of positive ions moving towards the cathode consists of alcohol and argon ions. Due to collisions between the two types of ions the argon ions will extract enough electrons from the alcohol ions to become neutralised before they reach the region of the cathode. As a result only alcohol ions will arrive in the vicinity of the cathode. If conditions in that region are such that the ionised alcohol molecule loses all its energy by impact on the cathode, secondary electrons will be created which initiate a parasitic discharge. But, in the normal case, the ionised alcohol molecule will be neutralised before it reaches the cathode itself. It follows a discussion on technical aspects of the production of Geiger counters. In conclusion halogen counters are discussed. The characteristics of halogen counters are shown in a table, partly reproduced below.

Characteristics of Certain Halogen Geiger-Muller Counters Manufactured at the Commissariat a l'Energie Atomique

	Type of counter and particles detected	
	3 G 12 : $-\gamma$	3 B 15 : $-\beta$ *With a longitudinal window*
Envelope and cathode material, and total thickness	Pyrex Glass and Internal Chromium-Iron Cathode 500 mg/cm²	Pyrex Glass and Internal Chromium-Iron Cathode 40 mg/cm²
Length in mm.	100	100
Counting threshold in volt.	350 ± 50	350 ± 50
Length of the operating range in volt.	≧ 150	≧ 150
Gas filling in cm. Hg	Neon: 20 Argon: 0.4 Bromine: 0.04	Neon: 20 Argon: 0.4 Bromine: 0.04
Service life in no. of counts	≧ 10^{10}	≧ 10^{10}
Counting rate in counts per min.	40 if shielded with 6 cm. lead	40 if shielded with 6 cm. lead
Dead time in μ	200	200
Time of restoration in μ	230	230
Operational temperature	−70°C to +100°C	−70°C to +100°C

Sommaire : Un article détaillé sur le projet et l'exécution des compteurs de Geiger-Muller.

Article by A. Benoit *Le Vide* 9, March 1954 1475-1491

55/IV

A Sealed-Off Metal Proportional Counter for Cosmic Ray Work

United Kingdom. The conventional selection system, employed to trigger cloud chambers and actuated by penetrating showers, consists of trays of Geiger counters connected to the chamber which will effect the triggering, if more than one counter in the tray is simultaneously fired by the ionising particles. This arrangement, and in particular the use of the normal Geiger counter, has been found unsatisfactory as two or more particles may pass through the Geiger counter without firing more than one counter. A new arrangement is reported which provides for the substitution of one of the Geiger counter trays with proportional counters of rectangular cross section. Each of the latter counters will operate only if the ionisation voltage in it exceeds a certain value and its output will then be put into coincidence with Geiger counter trays. The proportional counters were made from copper wave-guide tubing, $7.2 \times 3.3 \times 35$ cm. and 2 mm. thick. Brass end plates soldered to the tubes carried Pyrex glass seals which facilitated the suspension and connection of a centrally positioned tungsten wire of 0.004 inch diameter. After chemical cleaning the counter was pumped by a system containing a liquid air trap for 24 hours. During that period it was baked for 2 to 3 hours at 150°C. and as a result the ultimate pressure was 10^{-5} to 10^{-6} mm. Hg. Then a mixture of 14% methane and 86% argon was introduced at 65 cm. Hg pressure at 15°C. After circulating the gas mixture over hot calcium chips for 16 hours the counter was sealed off. Operating the counters with a feed back amplifier a gain of 10^5 was obtained. The new counters have not yet been employed in the proposed modified triggering system of cloud chambers but tests carried out on these counters in conditions simulating the modified arrangement have proved: (*a*) That the new counters can detect single ionising particles with nearly 100% efficiency, (*b*) that a stable gas multiplication of about 150 is obtained at a counter voltage of 2,200 and (*c*) that the response is independent of the distance between the path of the ionising particle and the centrally suspended wire. Finally the energy loss distribution at half-height was calculated to be 70% of the most probable energy loss.

Sommaire : Description d'un compteur proportionnel pour système de selection opéré par douches de rayons cosmiques passant au travers de la chambre, pour le control de chambres à ionisation dans les travaux sur les rayons cosmiques.

Article by J. S. Buchanan *J. Sci. Instrum.* 31, April 1954 136-137

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Reactions Between Oxide Cathodes and Gases at Very Low Pressures

56/IV

See Abstract No. : 144/I

Decay of Emission from an Oxide-Coated Cathode Due to Adsorption of Matter Liberated from the Anode

57/IV

India. Poisoning effects in valves using oxide-coated cathodes are generally attributed to the presence of oxygen, carbon monoxide, chlorine and carbon dioxide in the envelope of the valve. In spite of recent developments in the field of gettering techniques such effects still persist. Three performance phenomena characteristic of poisoning effects are mentioned: (*a*) When switching on the anode voltage, the current often shows an immediate decay which lasts for about 1 minute. On switching off the anode voltage, normal electron emission is restored after about the same time interval. (*b*) If micro-second pulsed voltage is applied instead of steady d.c. voltage, up to 100 times larger currents may be obtained which are attributed to a very sharp initial decay of current lasting no longer than 10^{-2} seconds, recovered subsequently. Finally, (*c*) symptoms of permanent decay of emission can also often be traced. There is evidence that the phenomena mentioned are related to the surface condition of the anode, and the author, taking this new hypothesis into account, gives a mathematical analysis of the conditions where matter liberated from the anode by electron bombardment is adsorbed on the cathode surface and causes the decay observed. Two cases are considered, mobile adsorption and immobile adsorption. The treatment is based on the theory of absolute reaction rates and formulae are developed for the determination of the time rate of adsorption—time rate of desorption (evaporation) for both conditions. Both equations can be solved by graphical means yielding in the case of mobile adsorption a value of about 10^{-4} to 10^{-2} seconds for the decay time rate. The values obtained indicate that the effective area for mobile adsorption is appreciably less than the geometrical area of the cathode and is most likely confined to the defective regions of the cathode surface. It is assumed that a freshly produced valve is evacuated to a pressure of 10^{-6} mm. Hg. At this stage the poisoning agent may be present at a partial pressure of 10^{-8} mm. Hg. If this value rises to, or above, the value of the operational pressure (10^{-6} mm. Hg.) symptoms of poisoning will occur. There is evidence that the poisonous particles are deposited on the anode surface during the activation period. Of the various gases and vapours present in the atmosphere of the valve, oxygen is the most likely to cause the poisoning phenomena observed. During activation the cathode reaches temperatures up to 1,500°K. In this process oxygen may be evolved by the dissociation of barium oxide according to:

$$2\ BaO \rightleftharpoons 2\ Ba + O_2$$

At 1,275°K, equilibrium pressure of oxygen for this reaction is 10^{-11}mm. Hg. At the same time additional quantities of free oxygen are evolved by electrolytic dissociation within the body of the cathode. It is possible therefore that in subsequent operation of the valve electron bombardment of the anode results in a sudden increase of the partial oxygen pressure reaching the operational vacuum, *i.e.* 10^{-6} mm. Hg. This value is above that for the equilibrium of oxygen in the cathode and rapid deposition on the cathode face becomes possible. In conclusion the author offers a tentative explanation for the decay phenomena observed when operating with a pulsed voltage, based on the results of his investigation.

Sommaire : On examine le procédé de déterioration des courants d'émission de cathode à oxyde.

Article by S. Deb *J. Brit. I.R.E.* **14**, April 1954 157-167

Results of Experiments on the Behaviour of Nickel for Cathodes, Heated under Vacuum

58/IV

See Abstract No. 143/I

Sintered Thermionic Cathodes formed from Nickel and Alkaline Earth Oxides

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France. Recent progress of powder metallurgy has facilitated the manufacture of cathodes by pressing and sintering. The authors report on the preparation and performance of oxide cathodes prepared from pure nickel powder and powder of barium and strontium carbonates in a proportion of 8 atoms of nickel to 2 molecules of $(CO_2)_3$ BaSr and, alternatively, of 9 nickel atoms to 1 molecule of $(CO_2)_3$ BaSr. After mixing and adding a binder in the form of paraffin dissolved in benzene the powder is compressed under a pressure of 7,500 kg/cm² into a bar 40 mm. long 2 mm. wide and 0.1 to 0.3 mm. thick. Sintering was originally carried out *in vacuo*. In the temperature range up to 900°C the carbonates decompose causing first the separate formation of BaO and SrO crystals and subsequently the formation of mixed crystals. Below 900°C degassing is high but above that temperature a vacuum of 10^{-5}mm. Hg can be maintained without difficulty. Sintering at a maximum temperature of 900°C gives a product showing little shrinkage but great fragility. At 1,100°C shrinkage is appreciable but fragility is improved. Exposure to atmospheric air may cause deformation of the sintered product and therefore the sintered cathode should be assembled into the valve immediately and the valve should be evacuated at once. Alternatively, sintering was carried out in a hydrogen atmosphere. In both cases the sintering time was fixed at about 1 hour. The sintered cathodes were assembled on a valve base of the type 807. All subsequent operations to complete the valve followed the normal processing procedure. During activation it was noticed that substantial activation could be obtained purely thermally. If purely thermal treatment is followed by treatment with the thermionic current switched on, a better activation is obtained at relatively low temperature than by purely thermal treatment at very high temperatures. The treatment just mentioned, if timed properly and carried out at the right temperature, yields a quality of emission which is sufficiently labile to obtain the desired final degree of activation by adjusting subsequent treatment accordingly. Generally speaking, the emission obtained after activation from sintered electrodes agrees well with the

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figures given in the literature for conventional oxide cathodes. It appears that sintered cathodes are less liable to be affected by poisoning agents. In a particular experiment, the sintered cathode was operated in an atmosphere of oxygen at a pressure sufficiently high to produce a glow but the emission of the cathode remained the same as that obtained in high vacuum. Operating with a pulsed voltage of about a micro-second duration the current could be increased ten times compared with operation at a steady d.c. voltage. Finally, the effect of changes in the production procedure of sintered cathodes, such as a change in the sintering temperature, on the performance of the cathode is discussed.

Sommaire : On indique d'abord la préparation des cathodes par compression et frittage d'un mélange de poudres de nickel et de carbonate double de baryum et de strontium. On compare enfin les résultats observés en faisant varier la température de frittage, l'atmosphère de frittage et les proportions des deux constituants.

Article by G. Mesward & R. Uzan *Le Vide* 9, March 1954 1492-1507

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Fused Vacuum-Tight, Metal-to-Ceramic, Ceramic-to-Glass, Metal-to-Glass and Metal-to-Mica Sealing by Powdered Glass Techniques 61/IV

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Fused Vacuum-Tight, Metal-to-Ceramic, Ceramic-to-Glass, Metal-to-Glass and Metal-to-Mica Sealing by Powdered Glass Techniques 62/IV

United States. A method is described of effecting vacuum-tight seals between a variety of materials which has been applied with success in the mass production of proportional or Geiger counters for low-energy β-rays and X-ray photons. The counters, about 4 inch. long and of a diameter of 1 inch consist of an outer envelope (the cathode) carrying a mica window at one end and joined to a piece of ceramic tubing at the other end which connects to, and forms an insulation for, the anode support at that end. The latter carries the exhaust stem. The choice of the materials to be employed in the construction of these counters is governed by two major factors: (*a*) Halogens form part of the gas filling of the counters. Therefore precautions must be taken to prevent corrosion. (*b*) A very thin mica window has to be used and this determines the coefficient of expansion of the materials, to which it is joined. The following materials have been found to be satisfactory: For the cathode and the anode support—stainless ferritic steel No. 446 containing 28% chromium. This material has a mean linear coefficient of expansion of approximately 105×10^{-7}/°C in the temperature range –40 to 500°C. During high temperature processing it acquires a film of chromic oxides which prevents scale formation. For the ceramic tube Fosterite was chosen which has a coefficient of expansion of approximately 97×10^{-7}/°C. The pumping stem is made from 0010 or 0120 glass. As a sealing material powdered glass Corning 7570, which wets mica and flows easily in oxidising atmosphere at 600° C, has been used in all cases. Allowance must be made for the fact that its coefficient of expansion is slightly lower than that of the ceramic used. Its softening point is below that of the glasses mentioned so that butt welding of glass tubing can be carried out without distortion of the latter. Before use the 7570 glass is pulverised in a ball mill until it passes through a 100–mesh screen. It is then mixed with distilled water and the paste thus formed is painted on to the parts which have to be sealed. After joining the components the assembly is placed in an oven and heat-treated in the temperature range of 550–600°C. This technique requires that the seals effected are of the compression type. It can only be used in tubes which permit firing in an oxidising atmosphere, as 7570 glass flows readily only in the presence of a sizable amount of oxygen.

Sommaire : Description d'une variété de composés pour sceller le métal à la céramique, au verre, au mica, par l'emploi des techniques du verre pulverisé.

Article by N. Anton *Ceramic Age* 63, June 1954 15-19

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A.B.M.	Associaco Brasiliera de Metais, Via Duto, Donna Paulina 80-8°, Andar (Palocio Maua), Sao Paulo, Brazil.
Acta Metallurg.	Acta Metallurgica, 57, East 55th Street, New York 22, N.Y., U.S.A.
Acta Phys. Austriaca	Acta Physica Austriaca, Springer Verlag, Mölkebastei 5, Vienna, 1, Austria.
A.E.C. Rep.	U.S. Atomic Energy Commission Reports, Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.
A.E.R.E. Rep.	U.K. Atomic Energy Research Establishment Reports, Her Majesty's Stationery Office, P.O. Box 569, London, S.E.1, England.
Amer. J. Med Sci.	American Journal of Medical Science, Lea & Febiger, Philadelphia, 6, Penn, U.S.A.
Amer. J. Phys.	American Journal of Physics, American Institute of Physics, Michigan State College, East Lansing, Michigan, U.S.A.
Analyst	The Analyst, The Society of Public Analysts, 7-8, Idol Lane, London, E.C.3, England.
Analyt. Chem.	Analytical Chemistry, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Ann. Phys. Paris	Annales de Physique, Librarie Maison et Cie, 120, Boulevard St. Germain, Paris 6, France.
Appl. Sci. Res.	Applied Scientific Research, Martinus Nijhoff, The Hague, Holland.
Ark. Zoologi	Arkiv for Zoologi, Almquist & Wiksells Boktryckeri AB, Stockholm, Sweden.
Brit. J. Appl. Phys.	British Journal of Applied Physics, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
Bull. Classe Sci.	Bulletin de la Classe des Sciences, Academie Royale de Belgique, Palais des Academies, 1, Rue Ducale, Bruxelles, Belgium.
Bull. Math. Biophys.	Bulletin of Mathematical Biophysics, University of Chicago Press, 5750, Ellis Avenue, Chicago 37, U.S.A.
Canad. J. Phys.	Canadian Journal of Physics, The National Research Council, Ottawa, Canada.
Canad. Mach. & Manufact. News	Canadian Machinery and Manufacturing News, Maclean-Hunter Publishing Co. Ltd., 418, University Avenue, Toronto, Ontario, Canada.
Ceramic Age	Ceramic Publishing Co. Inc., 421, Parker Street, Newark 4, N.J., U.S.A.
Chem. Engng.	Chemical Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Chem. Engng. Progr.	Chemical Engineering Progress, American Institute of Chemical Engineers, 120, East 41st Street, New York 17, N.Y., U.S.A.
Chem. & Industr.	Chemistry and Industry, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
Chem. Ing. Tech.	Chemie Ingenieur Technik, Verlag Chemie G.m.b.H., Weinheim/Bergstrasse, Haupstrasse 129, Germany.
Chem. Technik.	Chemische Technik, VEB Verlag Technik, Unter Den Linden 12, Berlin, N.W.7, Germany.
Compr. Air Mag.	Compressed Air Magazine, Compressed Air Magazine Co., Phillipsburg, N.Y., U.S.A.
C.R. Acad. Sci. Paris	Comptes Rendus des Séances de L'Academie des Sciences. Messrs. Gauthier-Villar, 55, Quai des Grands-Augustine, Paris, France.
Electronics	Electronics, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Endeavour	Imperial Chemical Industries Ltd., 26, Dover Street, London, W.1, England.
Engineer	The Engineer, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Eng. News Rec.	Engineering News Record, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Food Engng.	Food Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Food Technol.	Food Technology, Institute of Food Technologists, 119-123, West Park Avenue, Champaign, Ill., U.S.A.
Glas-und Hochvakuum Tech.	Glas und Hochvakuum Technik, Rudolf A. Lang Verlag, Leibnizstrasse 64, Berlin-Charlottenburg 4, Germany.

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Industr. Engng. Chem.	Industrial and Engineering Chemistry, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Inst. Chem. Engrs.	Institute of Chemical Engineers, 56, Victoria Street, London, S.W.1, England.
Inst. Refrig.	The Institute of Refrigeration, Dalmeny House, Monument Street, London, E.C.3, England.
Instruments	Instrument Publishing Co. Inc., 921, Ridge Avenue, Pittsburg 12, Pa., U.S.A.
Instrum. Mfg.	Instrument Publishing Co. Inc., 921, Ridge Avenue, Pittsburg, 12 Pa., U.S.A.
J. Appl Phys.	Journal of Applied Physics, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Brit. I.R.E.	Journal of the British Institution of Radio Engineers, British Institution of Radio Engineers, 9, Bedford Square, London, W.C.1, England.
J. Inst. Metals	Journal of the Institute of Metals, The Institute of Metals (Inc.), 4, Grosvenor Gardens, London, S.W.1, England.
J. Inst. Petrol	Journal of the Institute of Petroleum, 26, Portland Place, London, W.1, England.
J. Lab. Clin. Med.	Journal of Laboratory and Clinical Medicine, 3207, Washington Blvd., St. Louis, Missouri, U.S.A.
J. Metals	Journal of Metals, American Institute of Mining and Metallurgical Engineers Inc., 29, West 39th Street, New York 18, N.Y., U.S.A.
J. Opt. Soc. Amer.	Journal of the Optical Society of America, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J. Phy. Soc. Japan	Journal of the Physical Society, Japan. Faculty of Science, Tokio, Japan.
J. Res. Nat. Bur. Stand.	Journal of Research of the National Bureau of Standards, U.S. Government Printing Office, Washington 25, D.C., U.S.A.
J. Sci. Instrum.	Journal of Scientific Instruments, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
K. Fysiograf. Sällskapets Handlingar	Kungl. Fysiografiska Sallskapets Handlingar, Histological Institute, University, Lund, Sweden.
K. Fysiograf. Sällskapets I. Lund. Forh.	Kungl. Fysiografiska Sallskapets I Lund Forhandlingar, Histological Institute, University, Lund, Sweden.
Lab. Practice	Laboratory Practice, United Trade Press Limited, 9, Gough Square, Fleet Street, London, E.C.4, England.
Lancet	The Lancet Limited, 7, Adam Street, Adelphi, London, W.C.2, England.
Light Metals	Temple Press Ltd., Bowling Green Lane, London, E.C.1, England.
Machinery	Machinery Publishing Co., Clifton House, 83-117, Euston Road, London, N.W.1, England.
Mater. & Meth.	Material and Methods, 330, West 42nd Street, New York 18, N.Y., U.S.A.
Metal Finish.	Metal Finishing, Finishing Publications Inc., 11, West 42nd Street, New York 18, N.Y., U.S.A.
Metal Progr.	Metal Progress, The American Society for Metals, 7301, Euclid Avenue, Cleveland 3, Ohio, U.S.A.
Metallurgia	Metallurgia, The Kennedy Press Ltd., 31, King Street West, Manchester 3, England.
Mod. Refrig.	Modern Refrigeration, Refrigeration House, Victoria Road, Woking, Surrey, England.
Murex Rev.	Murex Review, Murex Ltd., Rainham, Essex. England.
Nature	Nature, Macmillan and Co., St. Martin's Street, London, W.C.2, England.
Naturwiss.	Naturwissenschaften, Springer Verlag, Jebenstrasse 1, Berlin-Charlottenburg, Germany.
Nucleonics	Nucleonics, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Oyo Butsuri	Journal of Applied Physics, The Society of Applied Physics, Faculty of Technology, University of Tokio, Japan.

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Philos. Mag.	Philosophical Magazine, Taylor & Francis Limited, 18, Red Lion Court, Fleet Street, London, E.C.4, England.
Phys. Rev.	Physical Review, American Institute of Physics, Brookhaven National Laboratory, Upton, Long Island, N.Y., U.S.A.
Power	Power, McGraw-Hill Publishing Co. Inc., 330, West 42nd St., New York 36, N.Y., U.S.A.
Proc. Inst. Electr. Engrs.	Proceedings of the Institution of Electrical Engineers, Institution of Electrical Engineers, Savoy Place, Strand, London, W.C.2, England.
Proc. Phys. Soc.	Proceedings of the Physical Society, The Physical Society, 1, Lowther Gardens, Prince Consort Road, London, S.W.7, England.
Proc. Roy. Soc.	Proceedings of the Physical Society, The Royal Society, Burlington House, Piccadilly, London, W.1, England.
Proc. Roy. Soc. Med.	Proceedings of Royal Society of Medicine, 1, Wimpole Street, London, W.1, England.
Proc. Soc. Exp. Biol. Med.	Proceedings of the Society for Experimental Biology and Medicine, College of the City of New York, New York, 31, N.Y., U.S.A.
Prod. Engng.	Product. Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Prod. Finish.	Product Finishing, Sawell Publication Limited, 4, Ludgate Circus, London, E.C.4, England.
Reader's Dig.	Reader's Digest Association Ltd., 27, Albemarle Street, London, W.1, England.
Refrig. Engng.	Refrigerating Engineering, American Society of Refrigerating Engineers, 40 W. 40th Street, New York 18, N.Y., U.S.A.
Rev. Mod. Phys.	Review of Modern Physics, American Institute of Physics, Lancaster, Pa., U.S.A.
Rev. Prat. Froid	Revue Pratique du Froid, 254, Rue de Vaugirard, Paris 15e, France.
Rev. Pure Appl. Chem.	Review of Pure and Applied Chemistry, The Royal Australian Chemical Institute, Kelvin Hall, 55, Collins Place, Melbourne, Australia.
Rev. Sci. Instrum.	Review of Scientific Instruments, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
Silikattech.	Silikattechnik, Verlag Technik G.m.b.H., Berlin, C, Oranienburger Strasse 13-14.
Stahl u. Eisen	Stahl und Eisen, Düsseldorf, Germany.
Stain Technol.	Stain Technology, Biotech Publications, Geneva, N.Y., U.S.A.
Steel	Steel, Penton Publishing Co., Penton Building, Cleveland 13, Ohio, U.S.A.
Suff. Tech. Pap.	Technical Paper, Suffield Experimental Station, Ralston, Alberta, Canada.
Sylvan. Tech.	Sylvania Technologist, Sylvania Electric Products, Inc., Sylvania Center, Bayside, N.Y., U.S.A.
Times Sci. Rev.	Times Science Review, The Times, London, E.C.4, England.
Trans. Am. Soc. Metals	Transactions of the American Society of Metals, American Society of Metals, 7301, Uclid Avenue, Cleveland 3, Ohio, U S.A.
Trans. Faraday Soc.	Transactions of the Faraday Society, The Faraday Society, Aberdeen University Press Limited, 6, Upper Kirkgate, Aberdeen, Scotland.
Univ. Calif. Rad. Lab. Rep.	University of California Radiation Laboratory Reports, University of California, Berkeley, California, U.S.A.
Vakuum-Tech.	Vakuum-Technik, Rudolf A. Lang Verlag, Leibnizstrasse 64, Berlin-Charlottenburg 4, Germany.
Le Vide	Le Vide, Société Française des Ingénieurs des Techniciens du Vide, 44, Rue de Rennes, Paris 6e., France.
World Refrig.	World Refrigeration, 181, Kensington High Street, London, W.8, England.
Zentralblatt	Zentralblatt für Bakteriologie, Parasitenkunde, Infektionskrankheiten und Hygiene, VEB Gustav Fischer Verlag, Jena, Germany.
Z. Phys.	Zeitschrift für Physik, Springer Verlag, Jebenstrasse 1, Berlin-Charlottenburg, Germany.

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